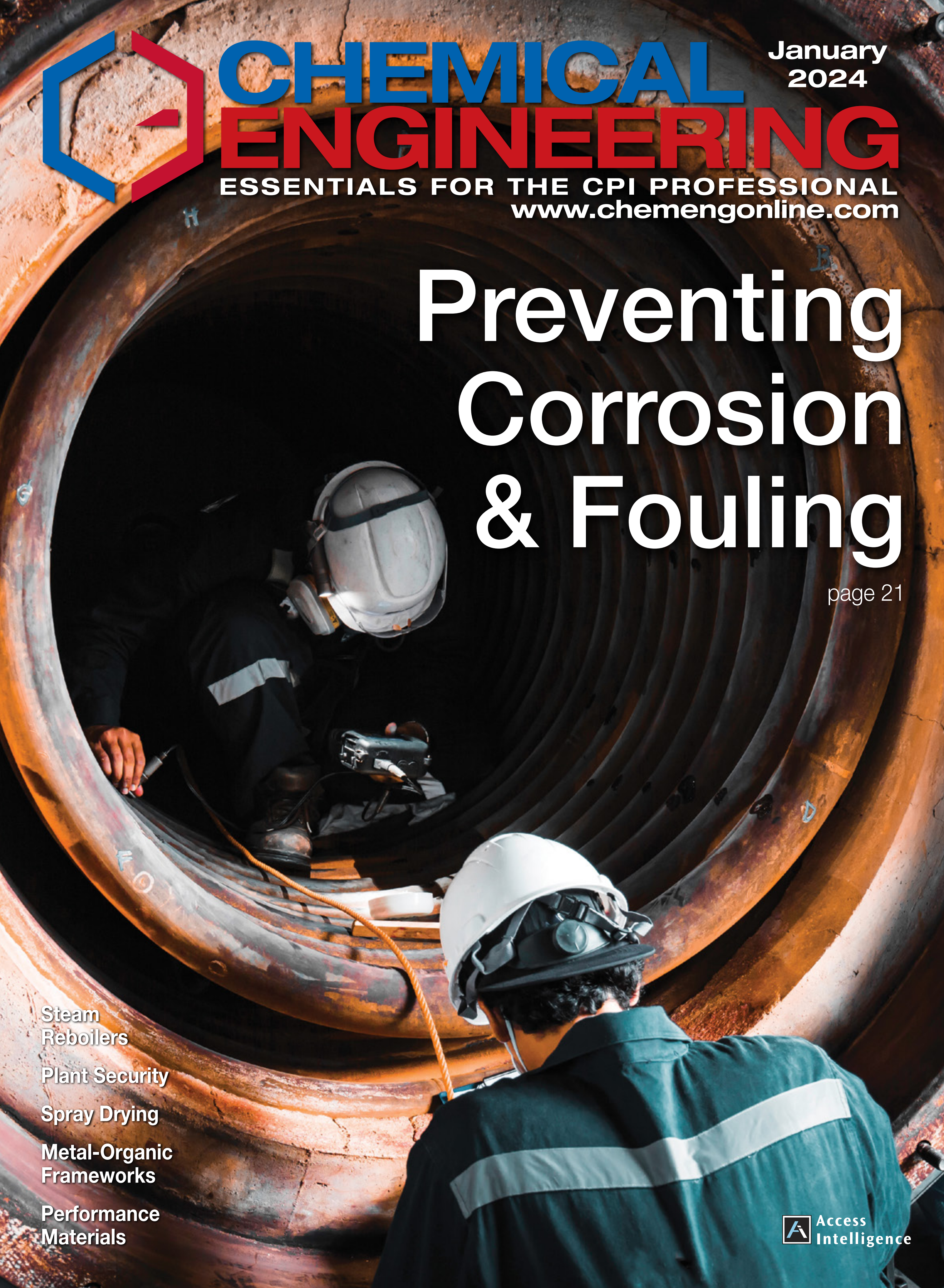

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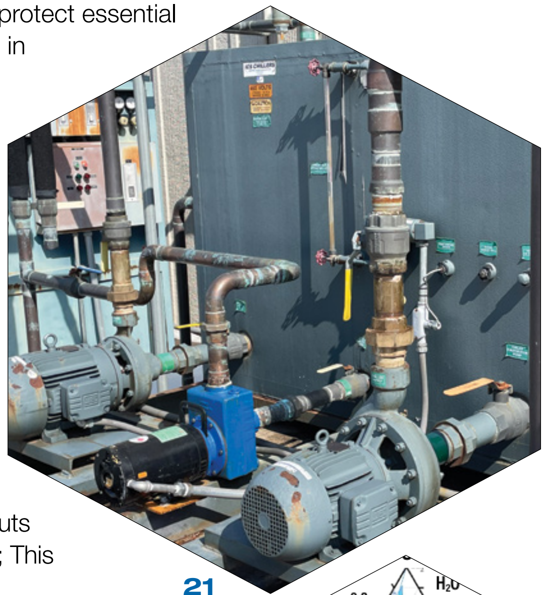

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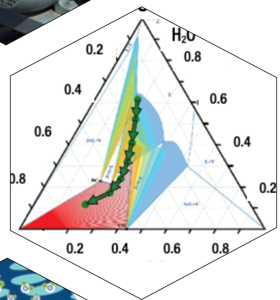

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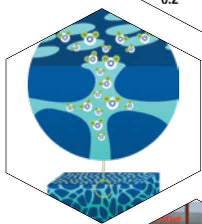

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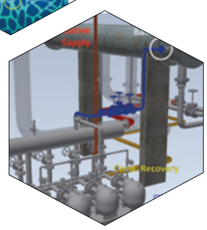

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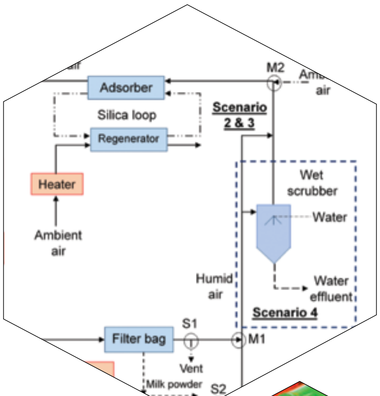

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CHEMICAL ENGINEERING
ESSENTIALS FOR THE CPI PROFESSIONAL

Editor's Page

CPI Outlook for 2024

Following a strong year for the chemical process industries (CPI) in 2022, 2023 saw modest growth, with global industrial output rising only 0.6% and chemical production growth at about 0.3% [*1*]. Numerous factors contributed to the lower demand for chemical products, including inflation and the de-stocking of very high inventory levels that resulted from critical supply-chain disruptions and over-ordering in the previous years.

In 2024, global industrial output is expected to pick up by 1.8% and global chemical production is expected to grow by about 2.9%, according to the American Chemistry Council's (ACC; Washington, D.C.; www.americanchemistry.com) year-end report [*1*]. The report also indicates that while an economic downturn is expected in the short-term, longer term prospects for U.S. chemical production are positive, with continuing energy advantages in the U.S., an expected modest increase in demand as high inventory levels are depleted, and initiatives to promote "clean" energy and domestic production.

Drivers

The ongoing energy transition is a strong driver for chemical demand. The move toward renewable energy sources and increasing decarbonization goals depend strongly on the CPI for materials and support. For example, while the ACC estimates that approximately $4,000 of chemical products are contained in an average automobile, electric vehicles (EVs) require even more chemistry-based components, such as high-performance polymers and battery components. And, EV sales are on the rise, accounting for a record 7.9% of total industry sales in the third quarter of 2023 according to Cox Automotive (www.coxautoinc.com).

In the U.S., government initiatives, such as the Infrastructure Investment and Jobs Act (IIJA), the Inflation Reduction Act (IRA), and the Creating Helpful Incentives to Produce Semiconductors (CHIPS) and Science Act, are driving investments into sectors that require materials from the CPI. The impact of these policies is expected to boost chemicals production in the long term.

CPI products play an important role in decarbonization goals to reduce emissions. In its 2024 Outlook [*2*] Deloitte (www2.deloitte.com) points out that "the chemical industry supports more than 75% of all emissions reduction technologies needed to meet net-zero goals by 2050," citing products such as battery materials, refrigerants for heat pumps and lubricants for wind turbines, as well as technologies for recycling, particularly for plastics and batteries. Deloitte expects these needs to increase demands for CPI products in 2024 as more projects come online.

Risks

The overall long-term outlook for the CPI is positive, with strong drivers as mentioned above. The ACC report reminds us of potential risks that could affect the expected outlook. Those risks include financial volatility, geopolitical conflicts, regulatory impacts and disruptive events, such as from weather. ■

Dorothy Lozowski, Editorial Director

1. American Chemistry Council, ACC Year-End Situation and Outlook, November 2023, www.americanchemistry.com.
2. Deloitte Research Center for Energy & Industrials, 2024 Chemical Industry Outlook, www2.deloitte.com.

Edited by:
Gerald Ondrey

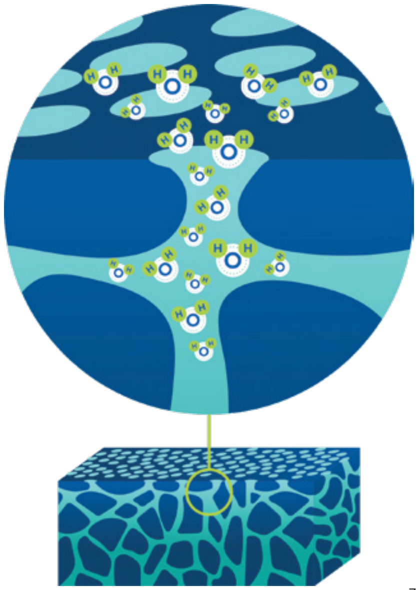
Fouling-immune membrane available for brackish-water RO applications

In November 2023, the first commercial test was initiated for a novel, fouling-immune membrane for brackish-water reverse osmosis (BWRO) applications through an early-access program. Membrane developer ZwitterCo (Woburn, Mass.; www.zwitterco.com) says its membrane is fouling-immune in cases where fouling of membranes by natural organic matter necessitates frequent membrane cleaning. By enabling restoration of membrane performance by simple water flushing, the new membrane reduces maintenance downtime and lengthens membrane lifespans.

The key aspect of the membrane is a specially engineered permanent barrier layer made from a proprietary co-polymer containing zwitterionic species. Zwitterionic compounds exhibit both positive and negative charges on the same molecule, which renders them highly hydrophilic. "The idea of harnessing zwitterions for water applications has been around for some time, but the challenge has been making a membrane with stable chemistry and with a solid structure, so that it can be used commercially," explains Jon Goodman, vice president for commercial development at ZwitterCo. "We've developed a co-polymer with zwitterionic species that has the required structure, but also prevents fouling by drawing water to the membrane surface so that organic molecules, such as FOG [fats, oils and greases], cannot adhere to it" (diagram).

The zwitterionic co-polymer acts as a barrier layer in a composite membrane that can be employed in high-fouling applications for BWRO, including secondary effluent from wastewater treatment systems and effluent polishing. The company points out that increasing demand for water reuse is driving the need for fouling-immune BWRO membranes.

The zwitterionic BWRO membrane follows a similar membrane that ZwitterCo previously launched for high-strength wastewater.



ZwitterCo

First commercial DAC facility in the U.S. captures CO_2 for concrete

In November 2023, Heirloom Carbon Technologies (Tracy, Calif.; www.heirloomcarbon.com) unveiled the United States' first commercial direct air capture (DAC) facility, which can capture up to 1,000 ton/yr of CO_2. The process first uses renewable energy to remove CO_2 from limestone in a kiln to make calcium oxide. The CaO is then hydrated to make calcium hydroxide powder, which is spread onto vertically stacked trays where, through maximized contact with air, it pulls CO_2 from the atmosphere, re-forming limestone ($CaCO_3$). This material is returned to the kiln, where the CO_2 is extracted, and the process begins again. By using easy-to-source materials like limestone, Heirloom says its technology is among the lowest-cost pathways to permanent CO_2 removal.

The captured CO_2 can be stored underground or embedded in concrete through a partnership with CarbonCure Technologies (Halifax, Nova Scotia; www.carboncure.com) (see *Chem. Eng.*, September 2015, pp. 18–24). Earlier this year, Heirloom and CarbonCure demonstrated the DAC-to-concrete CO_2 storage pathway that is being used to sequester CO_2 captured by the Tracy facility.

In August, Heirloom and partners were selected for one of the largest projects under the Department of Energy Regional DAC Hub program, with eligibility for up to $600 million in federal funding for a DAC facility in Louisiana. And in September, Heirloom and Microsoft announced a CO_2-removal deal in which Microsoft agreed to purchase up to 315,000 metric tons of CO_2 removal from Heirloom over a 10-year period.

CO_2 TO CO

SK Innovation Institute of Environmental Science & Technology, an R&D organization of SK Innovation Co. Ltd. (Seoul, South Korea; www.skinnovation.com), has successfully manufactured about 1 kg/d of CO using diatomic catalyst (DAC) technology, which selectively converts CO_2 into CO in an electrochemical process. The results of this demonstration were published in a recent issue of *Chemical Engineering Journal*.

According to the article, the diatomic catalyst consists of heteronuclear Ni and Fe sites anchored on N-doped carbon material (NiFe-DAC). A "unique coordination structure of hetero-diatomic site" enables the catalyst to preferentially convert CO_2 into CO with a CO selectivity of more than 97% under an industrially-relevant current density of 200 mA/cm^2 in 25-cm^2 and 140-cm^2 zero-gap membrane electrode assembly (MEA) CO_2 electrolyzers. A scale-up demonstration using a 140 cm^2 four-cell stack achieves an exclusive CO_2-to-CO generation rate of about 45 L/h.

The DAC technology was developed by the research team and published last year in *Applied Catalysis B: Environmental.*

'BLUE' H_2 STANDARD

Last month, TÜV SÜD AG (Munich, Germany; www.tuvsud.com) introduced TÜV SÜD CMS 77, a new standard for the certification low-carbon H_2 and blue H_2 and for its derivatives (currently ammonia). The CMS 77 standard sets a maximum threshold for greenhouse gas (GHG) emissions reduction potential allowed in the production process for H_2 and its derivatives to be considered as "low-carbon products." According to the standard, the GHG reduction of low-carbon H_2/blue

(Continues on p. 6)

H_2 and low-carbon ammonia/blue ammonia must be at least 70% compared to the global benchmark of 94 gCO_2eq/MJ_{LHV} (grams of CO_2 equivalent per megajoules, lower heating value). This corresponds to a GHG value of not more than 28.2 gCO_2eq/MJ_{LHV}. In addition to the claim of blue hydrogen/blue ammonia, the TÜV SÜD CMS 77 standard additionally requires the construction and use of facilities for carbon capture and geological storage with robust proof of permanency of geological storage.

Similar to the green-H_2 certification standard CMS 70, introduced in 2020, TÜV SÜD has defined the requirements and guidelines for the quantification and reporting of the carbon footprint of low-carbon H_2 and blue H_2 and its derivatives in a way that is consistent with international standards, such as ISO 14040, ISO 14067, ISO 27915 and ISO 17029.

PCL POLYMER

Sulzer Chemtech Ltd. (Winterthur, Switzerland; www.sulzer.com) is launching a new end-to-end licensed technology, Capsul, for the continuous manufacturing of polycaprolactone (PCL), a biodegradable polyester often used in the packaging, textile, agricultural and horticultural industries. The Capsul PCL technology includes all purification and polymerization steps as part of a fully integrated, highly efficient and continuous process. Building on Sulzer's expertise in separation and reaction processes, the new biopolymer technology is highly adaptable to a broad range of industrial scales that can help drive adoption of biodegradable and compostable PCL. Key applications for high-quality PCL include consumer packaging, 3D printing, footwear, agricultural films, textiles, and medical devices.

TDI PRODUCTION

Covestro AG (Leverkusen, Germany; www.covestro.com) is investing a mid- to high-double-digit million euro amount to modernize its toluene-diisocyanate (TDI) production plant in Dormagen, Germany, by 2025.

(Continues on p. 8)

Direct molten-salt electroplating cuts battery costs

The extremely high material purity demanded for conventional lithium-ion battery manufacturing creates considerable costs for transporting and refining lithium and other raw materials. Through the development of a single-step electroplating process that uses lower-purity raw materials and a nanostructured-foam electrode architecture, Xerion Advanced Battery Corp. (Kettering, Ohio; www.xerionbattery.com) aims to significantly decrease the costs, energy and carbon emissions in manufacturing batteries. Modeled after principles from the Hall-Héroult electroplating process used in aluminum refining, the company's DirectPlate technology combines purification, synthesis and deposition into a single step — requiring only molten salt to dissolve precursor materials, which can be directly deposited onto aluminum foil to create the electrode.

"We don't need battery-grade starting materials to make a high-quality, high-performance battery, because the electroplating process is inherently also a refining technique. We can use technical-grade, around 80% pure, lithium hydroxide or lithium sulfate. We've shown we can even use impure lithium chloride from brine sources or the mining concentrate itself from hard-rock processing," explains John Busbee, Xerion CEO and co-founder. Furthermore, once lithium is used in the molten-salt bath, silica can be isolated and used for the anode, and refined cobalt can be recovered as a valuable byproduct. Also, adds Busbee, since the molten-salt bath contains no organic materials, there is no need for recovery or re-distillation of the solvent, which is a significant source of energy consumption in traditional battery-manufacturing processes.

Xerion commissioned a lifecycle assessment for its manufacturing process, which showed that by using 80% lithium sulfate directly and avoiding traditional refining steps, the process realizes a 20–40% reduction in overall carbon emissions compared to other lithium-ion battery processes. Xerion has been running the process for several years at pilot scale, and work is currently underway to build out a new production line in Dayton, Ohio in early 2024, followed by larger plants in Dayton in late 2024 and a plant in Florida in 2026.

Converting natural gas to alcohols using MOFs

Capturing natural gas from petroleum drilling wells is economically challenging, but would have positive environmental impacts because it would eliminate the need for flaring and could allow the hydrocarbons to be converted into useful chemicals. Now, research led by chemists at the University of California at Berkeley (http://alchemy.cchem.berkeley.edu/home), in partnership with scientists from Lawrence Berkeley National Laboratory, Max Planck Institutes, Argonne National Laboratory, Northwestern University and several others, shows a potential way toward capturing value from natural gas that would otherwise be wasted. The technique uses metal-organic frameworks (MOFs) to convert methane and other components of natural gas into alcohols. The alcohols could be transported from wellheads as feedstock for other processes.

Using natural enzymatic oxygenation of carbon-hydrogen bonds as inspiration, the researchers developed MOFs whose reactive-site environments mimic that of an enzyme known as taurine alpha-ketoglutarate dioxygenase (TauD). Specifically, the MOF active sites react with O_2 to form high-spin Fe(IV)=O species that can oxygenate C–H bonds to generate alcohols.

"The high porosity and rigid crystalline structure of the MOFs allows easy entry of hydrocarbon gas molecules, allowing them to interact with the iron sites, which have a geometry around the iron center similar to the active site of the natural TauD enzyme," explains Jonas Börgel, a UC Berkeley postdoctoral fellow and first author of a recent *Science* paper discussing the work. The iron centers can activate oxygen at near-ambient temperatures to perform the oxygenation reactions in similar ways to the enzyme, he adds.

The research team is now focusing on the engineering aspects of the conversion, trying to determine a flow-reactor design that would allow natural gas and a co-factor (a source for necessary electrons in the reaction) to catalytically convert methane and ethane to methanol and ethanol in a continuous process. Börgel said that if it proves efficient at producing alcohols with less energy input than current processes, it might also be useful in large-scale facilities.

The 300,000-ton/yr plant was first commissioned in early 2015 and is considered to be one of the most advanced TDI production facilities in the world due to the use of the gas-phase technology developed by Covestro. Even before modernization, this plant requires up to 60% less energy and up to 80% less solvent than conventional processes.

The modernization work will include the installation of a reactor that will enable the reaction energy to be used for energy-efficient steam generation in the future. This will significantly improve the plant's energy efficiency and thereby reduce CO_2 emissions. After the modernization, the plant will consume up to 80% less energy than conventional processes for the production of TDI, and greenhouse gas emissions will be reduced by a further 22,000 ton/yr.

The modernization of the TDI plant in Dormagen is partly funded by the German Federal Ministry for Economic Affairs and Climate Action as part of the Federal Funding for Energy and Resource Efficiency.

WATER ELECTROLYSIS

In cooperation with Siemens Energy AG (Munich, Germany; www.siemens-energy.com), work on the water electrolysis facility at BASF SE's (www.basf.com) Ludwigshafen site — the so-called Hy4Chem-El project — is now entering the next phase of construction. With an output of 54 MW and a capacity of up to 8,000 metric tons (m.t.) of H_2 per year, the proton-exchange-membrane (PEM) electrolyzer will be one of the largest of its kind in Germany once it is operational. BASF and Siemens Energy plan to begin operating the water electrolysis plant in 2025.

Powered using electricity from renewable energy sources, the system will produce CO_2-free H_2 and thereby reduce greenhouse gas emissions at the site by up to 72,000 m.t./yr. BASF will primarily use this H_2 as a raw material in the manufacture of products with a reduced carbon footprint. In addition, the company will supply H_2 for mobility in the Rhine-Neckar Metropolitan Region to support the ramp-up of a H_2 economy in the area.

In cooperation with the State of Rhineland-Palatinate, the German Federal Ministry for Economic Affairs and Climate Protection is contributing up to €124.3 million to the project — up to €37.3 million of which will be financed by the gov-

(Continues on p. 9)

A continuous electrochemical process for direct-air CO_2 removal

Direct-air capture (DAC) of atmospheric carbon dioxide presents promise for helping to reach global-warming reduction goals. However, many DAC technologies are hindered by high costs and inefficiency.

RepAir Carbon (Yokne-am Illit, Israel; www.repair-carbon.com) recently launched a field prototype of its DAC technology, which is based on a continuous electrochemical process that lowers energy consumption and costs when compared to other DAC solutions. Similar to a fuel cell, RepAir's process employs two electrodes separated by a selective polymeric membrane. Cells can be "stacked" to multiply carbon-removal capacity.

"Atmospheric air is drawn into the cathode, where an electrical current generates hydroxide ions that bind to CO_2 molecules, forming carbonate and bicarbonate ions. Only these ions cross the membrane into the anode, wherein the binding process is undone, the hydroxides are consumed and pure CO_2 gas is drawn out," explains Amir Shiner, CEO and co-founder of RepAir Carbon. Unlike other DAC processes that must run in discrete batches, RepAir's technology can run continuously, because the cell is designed so that the electrode polarities and inlet airflow are swapped every few hours. Also, the technology can be fully run using renewable electricity with no additional heat source, which results in a 70% reduction in energy consumption when compared to other DAC processes, the company says.

Following the recently launched field prototype system, RepAir Carbon plans to introduce a larger 200-ton demonstration unit by 2025 and further expand to the industrial scale by 2027. "RepAir's straightforward modular design renders it easy to produce at a large scale, ensuring low capital expenditures. Furthermore, no waste or byproducts are produced," adds Shiner, noting that the core element of the cell is designed to last around ten years, after which all components can be disassembled for recycling.

This new stainless steel is ultra-resistant to corrosion

One of the major hindrances in saltwater electrolysis for "green"-hydrogen production is the cost of materials to construct electrolyzer components that can withstand an extremely corrosive saltwater environment. Currently, titanium coated with platinum or gold is usually required for such applications. However, a grade of stainless steel developed by researchers at the University of Hong Kong (HKU; www.hku.hk) exhibits much higher corrosion resistance than typical stainless steels, at a much lower cost than coated titanium.

The key to this new steel grade — called SS-H_2 — is a sequential dual-passivation mechanism that imbues additional corrosion resistance in stainless steel. Typical stainless-steel manufacturing employs a single-passivation method whereby the oxidation of chromium forms a protective film on the surface of stainless steel. The corrosion resistance of chromium-passivated steel becomes limited at potentials around 1,000 mV, which is significantly lower than what is required for water oxidation, meaning that such traditional stainless steels are not suitable for water-splitting applications.

The new sequential dual-passivation method includes a secondary manganese film layer on top of the chromium-containing film. This manganese-focused approach was quite novel. According to HKU mechanical engineering professor Mingxin Huang, who led the research, "the prevailing view is that manganese impairs raw corrosion resistance of stainless steel." However, he says, numerous atomic-level experimental results clearly indicated the passivation behavior of the steel's manganese content in a highly corrosive chloride environment at potentials up to 1,700 mV. Essentially, the primary Cr passivation layer protects at lower potentials, while the manganese layer provides additional protection at higher potentials, where the chromium layer can collapse and cause transpassive corrosion.

The university has partnered with an industrial factory to produce several tons of wire based on SS-H_2, and patents have already been granted in two countries, with several other applications pending.

Sand-based thermal-energy storage

Using concentrated solar thermal (CST) energy to supply industrial-heating requirements is a promising way to reduce the use of fossil fuels. This is the aim of Magaldi Green Energy (Rome, Italy; www.magaldigreenenergy.com), which developed its Solar Thermo Electric Magaldi (STEM) CST technology. STEM-CST not only converts solar radiation into thermal energy, but integrates energy storage in order to supply process steam, even in the absence of sunlight.

The patented STEM-CST technology consists of a primary-mirror field (heliostats) and a secondary reflector positioned over a ground-mounted solar receiver, consisting of a fluidized bed (FB) of silica sand. The FB can be uniformly heated to 600°C, and can then release the stored energy via heat exchangers immersed in the sand. This approach has the advantage over alternative molten-salt storage systems, because the sand can operate at both lower and higher temperatures. The STEM-CST system can deliver thermal energy within the required temperature range (100–400°C) for many industrial sectors, including paper, food-and-beverages, chemicals and plastics.

The first experimental module started up in June 2016 at the Integrated Energy Hub of A2A Enerie Future in San Filippo del Mela in Messina, Sicily, where it operated for 12,000 h.

In parallel, the Magaldi Group has developed MGTES (Magaldi Green Thermal Energy Storage), which is also based on a FB of sand, but uses electrical heating (from photovoltaic (PV) or wind generators) to "charge" the battery. Last March, Enel-X (Rome; www.enelx.com) and the Magaldi Group joined a partnership to further develop MGTES technology. The first application will involve supplying "green" thermal energy to meet the energy needs of vegetable-oil producer I.GI S.r.l. (Buccino, Italy), a supplier of Ferrero Group. It involves building a 5-MW PV plant and a 125-ton MGTES system with a daily storage capacity of 13 MWh of thermal energy. The MGTES system will become operational in the second half of 2024.

Converting a steel mill to climate-neutral steel production

To decarbonize steel production and its high carbon dioxide emissions, Fraunhofer researchers, TS Elino GmbH (Dürren; www.elino.de) and Salzgitter AG (all Germany; www.salzgitter-ag.com) are working on converting an existing steel mill to climate-neutral production methods. The aim is to produce steel by the direct reduction of iron ore with hydrogen, which would completely replace coke as a reducing agent. The H_2 required for this method is produced by electrolysis with electricity generated from renewable energy sources. Overall, this could reduce CO_2 emissions by up to 97%.

Work has already been underway for years to develop new technologies to decarbonize production. As part of the new BeWiSe project, which is funded by the German Federal Ministry of Education and Research (BMBF: Bonn, Germany), the Fraunhofer Institute for Ceramic Technologies and Systems (IKTS; Dresden, Germany; www.ikts.fraunhofer.de), partner institutes Fraunhofer Institute for Systems and Innovation Research (ISI; Karlsruhe) and Fraunhofer Institute for Environmental, Safety and Energy Technology (UMSICHT; Oberhausen) and Salzgitter are focusing on H_2-based direct reduction.

The project is studying the use of waste heat to increase the electrical efficiency of electrolysis using high-temperature electrolysis based on solid-oxide electrolysis cells (SOEC) developed at Fraunhofer IKTS. The partners are looking to optimize the entire process chain in terms of resource and energy efficiency. For this purpose, a direct-reduction demonstration plant (about 30-m tall) on the Salzgitter AG premises is being used.

"We have been working successfully with the Fraunhofer researchers for six years to transform steel production," says Alexander Redenius, head of Resource Efficiency and Technology Development at Salzgitter Mannesmann Forschung. "The direct reduction demonstration plant enables us to optimize the reduction process and how it interacts with the other process steps. Through this work, we are creating the basis for sustainable steel production with low CO_2 emissions." The company aims to convert one third of its steel production to the climate-friendly process with hydrogen as early as 2026. ■

ernment of Rhineland-Palatinate.

To put these numbers in perspective, BASF currently uses around 250,000 m.t./yr of H_2 at its Ludwigshafen site.

FCC CATALYST

W. R. Grace & Co. (Columbia, Md.; www.grace.com) recently launched Paragon fluid catalytic cracking (FCC) catalyst technology to help petroleum refiners produce transportation fuels with a lower carbon footprint. Paragon FCC catalyst incorporates a rare earth-based vanadium trap into high matrix-surface-area catalysts for the FCC unit. With Paragon technology, refiners can widen their FCC operating window and increase flexibility to process a range of feedstocks for greater profitability, says the company. Importantly, this technology leads to maximum bottoms upgrading of feedstocks, along with improved conversion at constant coke yield, allowing refiners to produce fuels in a more sustainable manner, Grace adds.

Paragon catalyst is the result of a multi-year R&D program to develop an advanced vanadium trap and builds on the metals tolerance of Grace's popular Midas catalyst platform.

USING ORGANIC WASTE

Alfa Laval AB (Lund, Sweden; www.alfalaval) and Bisviridi Ltd. (London, U.K.; bisviridi.com) are collaborating to elevate the sustainability potential of organic and food waste recycling. The partnership integrates Alfa Laval's Prodec Oil Plus decanter, designed for efficient oil separation, with anaerobic digestion (AD) to convert oil and fats waste into biofuel. Developed and patented by Bisviridi, this process produces Bio Crude, which can be refined into sustainable aviation fuel (SAF).

Prior to methanogenesis, the Prodec Oil Plus decanter efficiently extracts oil from organic waste, removing oils, fats and grease content. This allows the remaining components to be reintroduced into the AD system with minimal impact on the biogas-production process. The resulting Bio Crude boasts impressive purity levels of up to 99.5%, making it a suitable feedstock for petroleum refineries to produce biofuel for the SAF market. The AD plant can produce up to 300 L/h (or more) of oil. ❐

LINEUP

- BASF
- BOREALIS
- CEPSA
- CF INDUSTRIES
- EASTMAN
- INEOS
- KEMIRA
- LHYFE
- LINDE
- LYONDELLBASELL
- RÖHM
- SABIC
- SIKA
- SOLVAY
- TRINSEO
- UMICORE

Plant Watch

Trinseo inaugurates new PMMA depolymerization plant in Italy

December 7, 2023 — Trinseo (Berwyn, Pa.; www.trinseo.com) inaugurated a new polymethyl methacrylate (PMMA) depolymerization plant in Rho, Italy. This depolymerization operation will be designed to enable the efficient recycling of end-of-life PMMA, as well as other difficult-to-recycle materials, which will ultimately be used to produce acrylic resins, sheets and compounds containing recycled materials.

Umicore announces groundbreaking for fuel-cell catalyst plant in China

December 5, 2023 — Umicore N.V. (Brussels, Belgium; www.umicore.com) broke ground in Changshu, Suzhou, China for its large-scale fuel-cell catalyst plant. The new plant is expected to become the world's largest catalyst-production facility for proton-exchange membrane (PEM) fuel cells when it becomes operational in early 2026.

Linde increases hydrogen production capacity in Alabama

December 5, 2023 — Linde plc (Guildford, U.K.; www.linde.com) announced that it has increased the liquid hydrogen production capacity at its facility in McIntosh, Ala. Linde's McIntosh facility will now produce up to 30 metric tons per day (m.t./d) of liquid hydrogen for the local merchant market. Linde invested approximately $90 million in the project.

Lhyfe to build "green" hydrogen plant in northwest France

November 27, 2023 — Lhyfe (Paris, France; www.lhyfe.com) plans to develop a "green"-hydrogen production and distribution site at the Nantes Saint-Nazaire Port in France. As part of this project, Lhyfe will build an industrial unit with a production capacity of up to 85 m.t./d of green and renewable hydrogen (installed electrolysis capacity of 210 MW). This site is expected to be operational by 2028.

Kemira to expand production capacity for ferric sulfate in the U.K.

November 27, 2023 — Kemira Oyj (Helsinki, Finland; www.kemira.com) is executing a significant capacity expansion for ferric-sulfate-based water-treatment chemicals at a production site in Goole, U.K. The additional 70,000 m.t./yr capacity extension helps meet demand for coagulants used to regulate nutrient discharge in wastewater-treatment operations. The new capacity is set to begin operation in 2025. Kemira currently produces over 350,000 m.t./yr of water-treatment chemicals and other types of products at its four manufacturing sites in the U.K.

Cepsa begins first production of circular phenol from single-use plastics

November 27, 2023 — Cepsa (Madrid, Spain; www.cepsa.com) began its first production of phenol and acetone from recycled single-use plastic materials at the La Rábida Energy Park in Huelva, Spain. To carry out this first test, 300 tons of waste plastic were processed, providing an alternative raw material to petroleum.

Sika to expand polymer production plant in Texas

November 27, 2023 — Sika AG (Baar, Switzerland; www.sika.come) plans to invest in its polymer production site in Sealy, Tex. The company has initiated this expansion due to the rising demand for its concrete admixture products in the U.S. and Canada.

BASF increases defoamer capacity at its Dilovasi plant in Turkey

November 27, 2023 — BASF SE (Ludwigshafen, Germany; www.basf.com) announced the expansion of defoamer capacity at its Dilovasi plant in Turkey. Defoamers are used in the production of paints, coatings and inks.

Mergers & Acquisitions

Solvay completes spinoff of specialties business unit

December 11, 2023 — Solvay S.A. (Brussels, Belgium; www.solvay.com) has successfully completed the spinoff its specialties business activities into standalone firm Syensqo. Syensqo's product offerings include amines, composites, lithium derivatives, phenols, phosphorus specialties, solvents, surfactants and more.

Ineos to acquire LyondellBasell's ethylene oxide and derivatives business

December 8, 2023 — Ineos Ltd. (London; www.ineos.com) announced an agreement with LyondellBasell Industries N.V. (Rotterdam, the Netherlands; www.lyondellbasell.com) to buy its Ethylene Oxide and Derivatives business, including the Bayport Underwood site in Texas, for $700 million. The deal includes the 420,000-m.t./yr ethylene oxide plant, the 375,000-m.t./y ethylene glycols plant and the 165,000-m.t./y glycol ethers plant, together with all associated third-party business on the site. Ethylene oxide is a key raw material used in the production of pharmaceuticals, cosmetics, semiconductors, polyester, food packaging, construction materials and more.

Cepsa and C2X set up joint project to develop green methanol plant in Europe

December 7, 2023 — Cepsa and C2X, an independent company majority owned by A.P. Moller Holding, announced a joint plan to develop a "green" methanol plant in the

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port of Huelva. The project's aim is to reach an estimated production capacity of 300,000 m.t./yr of green methanol with a maximum production capacity of 380,000 m.t./yr. A final investment decision for this project, which would entail a cost of up to €1 billion, is expected to be made in 2025. Green hydrogen produced at Spain's Andalusian Green Hydrogen Valley, currently under development, would supply the green methanol plant.

Eastman completes sale of Texas City assets to Ineos

December 4, 2023 — Eastman Chemical Co. (Kingsport, Tenn.; www.eastman.com) announced that it has completed the previously reported sale of its Texas City operations to Ineos Acetyls for a total sale price of $490 million. The Texas City site was previously part of Eastman's Chemical Intermediates segment. Eastman has retained ownership of its plasticizer business at the site, which Ineos now operates for Eastman as part of this agreement.

Kemira to divest its oil-and-gas business for $280 million

December 4, 2023 — Kemira agreed to divest its oil-and-gas portfolio to Sterling Specialty Chemicals LLC, a U.S. subsidiary of Artek Group, a global industrial chemicals group based in India. The approximately $280-million transaction involves manufacturing facilities in the U.S., the U.K. and the Netherlands.

CF Industries completes acquisition of ammonia plant

December 4, 2023 — CF Industries Holdings, Inc. (Deerfield, Ill.; www.cfindustries.com) closed its acquisition of Incitec Pivot Ltd.'s (IPL) ammonia production complex located in Waggaman, La. Under the terms of the agreement, CF Industries purchased the Waggaman ammonia plant and related assets for $1.675 billion. The companies allocated approximately $425 million of the purchase price to a long-term ammonia-offtake agreement under which CF Industries will supply up to 200,000 m.t./yr of ammonia to IPL's Dyno Nobel subsidiary.

Borealis to acquire plastics recycling firm in Bulgaria

November 30, 2023 — Borealis AG (Vienna, Austria; www.borealisgroup.com) signed an agreement for the acquisition of Integra Plastics AD, a specialist in advanced mechanical recycling based in Bulgaria. Integra Plastics AD operates a mechanical recycling plant built in 2019, which can transform post-consumer waste into high-quality polyolefin recyclates.

Röhm and Sabic merging acrylics and polycarbonates businesses

November 27, 2023 — The Acrylic Products business of Röhm GmbH (Darmstadt, Germany; www.roehm.com) and Sabic's (Riyadh, Saudi Arabia; www.sabic.com) Functional Forms business unit will be merged to form a new firm called Polyvantis. Polyvantis will operate mainly in the acrylics and polycarbonate film and sheet industries, serving a multitude of segments, including building and construction, transportation and aviation and electrical and electronics. ■

Mary Page Bailey

Cybersecurity Threats Prompt Proactive Approach

An evolving threat landscape for plant security is attracting more attention to the development of operational technology (OT)-specific cybersecurity programs, underlining the importance of collaboration and blurring the line between cyber- and physical security

Cybersecurity threats against critical infrastructure sites, including chemical process industries (CPI) facilities, have been expanding in frequency, magnitude and sophistication. CPI sites are among those being targeted specifically because of vulnerabilities related to several factors, including large numbers of employees, suppliers and third-party contractors, as well as complicated cybersecurity protection requirements. Meanwhile, the ongoing digital transformation and greater connectedness of plant assets has meant a closer integration of traditional information technology (IT) systems with plant operational technology (OT) systems. This integration opens a much larger possible attack surface for cyber actors to exploit.

Greater cyber risk, coupled with the unique requirements of OT-system security, has spurred the industry to shift toward a more proactive approach to cyber protection that faces the changing threat landscape by focusing on the unique characteristics of OT cybersecurity to create an OT-specific cybersecurity program. While the cybersecurity risks for OT environments are growing, significant progress is being made.

Evolving threat landscape

In decades past, OT systems were largely isolated, running proprietary control protocols and using specialized hardware and software. With the acceleration of the digital transformation, OT systems are increasingly adopting IT solutions to enable connectivity for process optimization and remote access capabilities. IT-standard computers, operating systems and network protocols are more integrated with OT environments, supporting a broad range of capabilities, but also complicating the task of securing OT systems.

"Nation-state actors and cybercriminals are increasingly targeting operational technology systems used in critical infrastructure sectors," explains Eran Fine, CEO of NanoLock Security (Hod Hasharon, Israel; www.nanolocksecurity.com). The IT/OT convergence has introduced "vast new cyber risk exposure that organizations are still struggling to fully grasp and mitigate," Fine says. "It demands rethinking how cybersecurity tools, controls and processes are implemented to maintain both information security and operational resilience."

In leveraging the convergence between IT and OT systems, cyberthreat actors have grown their capabilities to carry out disruptive attacks on vital services, representing a concerning shift from previous years, Fine remarks.

Greg Hatcher, co-founder of White Knight Labs (Grand Rapids, Mich.; www.whiteknightlabs.com) confirms the cybersecurity challenge of IT/OT convergence: "Most cyberattacks are initiated from the IT space, and then attackers pivot to OT," says Hatcher. "And companies generally lack network segmentation, which is needed to prevent hackers from moving from the IT system to the OT network."

A recent study commissioned by Rockwell Automation and conducted by Cyentia Institute supports this, finding that over 80% of a set of 122 cyberincidents involving OT and industrial control systems (ICS) started with an IT-system compromise attributed to increasing interconnectivity across IT and OT systems and applications.

FIGURE 1. The IT/OT convergence has enlarged the possible surface area for cyberattacks, with most attacks initiated through the IT system

"Ransomware attacks have really taken off in terms of frequency over the past year," comments Mackenzie Morris, senior industrial consultant at Dragos Inc. (Hannover, Md; www.dragos.com), so organizations are becoming more proactive about cybersecurity to get ahead of the curve. "Over the past year or so, we have been seeing an increasing commoditization of cyberattacks, with ransomware-as-a-service being offered by cybercriminals and many more smaller groups getting involved," Morris says.

"The unfortunate reality is that it's no longer a matter of 'if' a company will get attacked; it's a matter of 'when,'" says Tom Cottle, principal functional safety and cybersecurity consultant at AcuTech Consulting Group (Vienna, Va.; www.acutech-consulting.com). He points out that malware and attack strategies show an increasingly sophisticated understanding of OT-specific knowledge on the part of cyber-adversaries.

Not all cyberthreats originate outside the perimeter, however. The threat of insider attacks is growing in importance, especially among CPI facilities, because of the large network of suppliers, employees and

independent contractors working on globally dispersed sites.

"Insider threats represent one of the most significant vulnerabilities facing industrial and critical infrastructure cybersecurity today," NanoLock's Eran Fine says. Insider attacks can be the result of intentional malicious actions, stolen credentials or even mistakes, Fine explains, but they have the potential to be highly damaging because insiders are likely have knowledge that those outside a company would not.

NanoLock recommends that organizations address credential abuse by monitoring and managing permissions, keys and passwords among employees and third-party contractors. "Privileged users, in particular, require additional safeguards and oversight to prevent unauthorized access, and insider privilege exploitation by contractors is an obstacle that must be overcome," Fine says.

One additional challenge for OT cybersecurity is the compatibility of legacy devices and software with modern security tools. Older legacy systems may not be able to support protections designed for modern IT systems, Fine says.

A positive sign in addressing these concerns is that companies have seemed much more willing to collaborate with peer organizations in this area than has been the case in the past. "Cooperation among organizations in cybersecurity has really accelerated recently," Dragos' Morris says. "Especially when it comes to participation in ISACs [information sharing and analysis centers] that are specific to industrial-sector cybersecurity. This type of collaboration allows organizations to share threat intelligence and quickly paint an overall picture of industrial cyberthreats."

An example of this is Dragos' Neighborhood Keeper Collective Defense System, a free, opt-in, anonymized information-sharing network available to all those using Dragos' cybersecurity platform for OT/ICS. Neighborhood Keeper is capable of detecting supply-chain risks and equipment threats, acting as a sort of collective defense while enabling industry and government partners to leverage the system as a cyber national-broadcasting service.

"Companies are much more willing to share tactics through third-party reporting organizations," says Tom Cottle from Acutech. "The message — that companies need to 'play nice' with each other in this area — seems well received," says Cottle. At one time, collaboration on process safety was a challenge, but now there is substantial cooperation on safety concerns — "the same will happen for cybersecurity," he says.

Programmatic approach

A response to increased cybersecurity risk and increased cybersecurity regulatory attention (see box, p. 14) on the part of CPI companies and other industrial entities, has been to adopt a more proactive strategy that aims to build a cybersecurity program specifically tailored to the characteristics of industrial OT systems.

James Goosby, executive-in-residence at the McCrary Institute at Auburn University (Auburn, Ala.; www.mccrary.auburn.edu), says over the past couple of years, "There seems to be a shift to a more proactive, ownership-based approach to OT cybersecurity." It's not optimal to holistically farm out OT cybersecurity to outside entities, Goosby says, because robust cybersecurity requires a close and detailed understanding of the assets and systems you are trying to secure. "To do that, you need a programmatic approach to cybersecurity risk mitigation," he notes. "It's not enough to rely on ones and zeros — you have to understand where all of your cyber assets are, which assets are connected to the network, as well as how those connections are made, and for what purpose."

Chuck Tommey, digital connectivity executive at Siemens AG (Munich, Germany; www.siemens.com) agrees: "Companies need to make sure they have an OT-specific cybersecurity program. Many still don't," he says. A majority of companies have some cybersecurity-related tools in place, but lack an overall, holistic plan, and lack the personnel resources to execute it (see box, this page).

Tommey echoes the analogy between safety and cybersecurity: "Companies need a programmatic approach to OT cybersecurity that mirrors the one they have for safety. All industrial companies have a continuous improvement mindset for safety — the same thing needs to happen for cybersecurity." Industrial facilities need to develop a culture of cybersecurity responsibility, Tommey says, including awareness training, disposal of used equipment and other areas.

OT CYBER WORKFORCE ISSUES.

One of the positive developments in OT cybersecurity has been the broader recognition in recent years of the OT cybersecurity community — individuals who have demonstrated knowledge and experience with operational systems and are focused on the unique needs of industrial control system (ICS) cybersecurity. However, there remains a stark need to bolster the ranks of this profession.

McCrary's Goosby says the need is great for more professionals in this area who have expertise with OT systems and relevant cybersecurity practices. "There is definitely a workforce issue, where we need to publicize information about these career paths and how to access them," he opines.

Tom Cottle, of AcuTech Consulting, agrees: "There is a real need for people with expertise both in control systems and cybersecurity to start to remedy workforce issues." There has been more interaction between IT and OT in recently, and there is a growing recognition that IT and OT need to be integrated, especially when it comes to cybersecurity, Cottle says. Efforts to integrate IT and OT cybersecurity through shifts in organizational alignment are ongoing and improving, he adds.

"Folks with engineering backgrounds and also training in cybersecurity are rare, so most people working in this area have just fallen into it in more haphazard way, Dragos' Morris says. "There's no proper pipeline for education in this area at the moment, so there's definitely a massive expertise gap. "What we have seen bear the most fruit is for companies to take young engineers and train them in cybersecurity," Morris says.

The McCrary Institute is furthering its development of cyber-informed engineering strategies through a partnership with Tuskeegee University (Tuskeegee, Ala.; www.tuskeegee.edu). The university is fostering awareness of CIE and how it can be applied.

"We want to develop research opportunities with suppliers to bring them into the lab environment and inform the curriculum," McCrary's Goosby explains. "We are seeking to leverage knowledge in a collaborative fashion and teach students about what tools are currently used in real-world industry settings." ❑

CYBERSECURITY REGULATIONS AND STANDARDS UPDATES

As cyberthreats intensify, governments are wrestling with how to regulate cybersecurity and how to establish and enforce requirements for cybersecurity programs in a diverse field of industry sectors.

"Until recently, cybersecurity standards were essentially voluntary for operators of critical infrastructure," explains NanoLock's Eran Fine. "New regulations now call for the adoption of security measures more specific to OT assets and critical infrastructure. They explicitly require proactive, preventative security measures, such as zero-trust and device-level protections, rather than just detection controls," says Fine.

In September 2023, NIST finalized a new revision of its special publication "Guide to Operational Technology Security" (NIST SP 800-82r3), which advocates "developing security policies, procedures, training and educational material that apply specifically to the OT system." Among other items, the standard advocates separate authentiication mechanisms for OT and corporate networks, restricting physical access to OT devices and assets and restricting unauthoriized modifiication of data.

In July 2023, the U.S. Securities and Exchange Commission (SEC; www.sec.gov) issued rules to enhance and standardize disclosures regarding cybersecurity risk management, strategy, governance and incidents by public companies. Meanwhile, in Europe, an update to cybersecurity directive 2022/2555, known as NIS-2, significantly expands the types of organizatons within its scope, including manufacturers of chemicals, food processors and makers of medical devices. The law requires organizations to take technical, operational and organizational measures to manage risks to their network and information systems, and to minimize the impact of potential incidents.

The scope of standards and regulations for industrial cybersecurity seems to be widening in terms of the organizations involved, reflecting an acknowledgement that industrial cybersecurity must be more far-reaching than in the past. Siemens' Chuck Tommey notes that transportation regulatory agencies are now thinking about industrial cybersecurity because of the far-reaching effects of a cyberattack on pipelines and shipping infrastructure (and more). Environmental agencies are looking at the potential implications of cyberattacks on air- and water-monitoring systems, The U.S. Coast Guard is looking at cybersecurity at ports and navigable rivers from the perspective of shipping resources and materials. ❑

As a path to begin operationalizing OT cybersecurity, "there has to be a board-level person who 'owns' OT cybersecurity," Tommey continues. For example, someone should have the responsibility of maintaining and updating the OT cybersecurity program, once developed, and the OT security program needs an executive-level sponsor who can collaborate with IT personnel. There should be a cyberincident-response plan, and of course, Tommey continues, the program needs a budget.

Programmatic approaches include not only prevention of intrusions, but also response to security incidents. "Only about half of all companies in this space have an incident-response plan that is focused on the ICS and OT," says White Knight Labs' Hatcher. "Some have tried to 'cut-and-paste' IT-centric response plans, and use them in the OT space, but that generally does not work well," Hatcher says.

Zero-trust

One cybersecurity concept that has been receiving a good deal of attention recently is that of "zero-trust" approaches to OT cybersecurity. Zero-trust refers to a cybersecurity paradigm that is focused on resource protection and that is based on the premise that trust must be continually evaluated, rather than implicitly granted. According to the U.S. National Institute of Standards and Technology (NIST; Gaithersburg, Md.; www.nist.gov), zero-trust architecture "is an end-to-end approach to enterprise resource and data security that encompasses identity, credentials, access management, operations, endpoints, hosting environments and interconnecting infrastructure." NIST says the initial focus should be on restricting resources to those with a need to access, and grant only the minimum privileges (such as read-only, write, delete) needed to perform the mission.

Zero-trust approaches represent a departure from previous network-protection schemes that focused on perimeter defense and where authenticated subjects are given authorized access to a broad collection of resources once on the internal network, NIST explains. In this situation, unauthorized lateral movement within the environment is a significant challenge.

"Zero-trust architectures that verify all users, limit access and protect critical data and functions are essential," according to NanoLock. "There has been a growing push recently to take a device-level, zero-trust approach focused on prevention, rather than just detection," NanoLock says. "OT networks differ in plant facilities and industries. OT security measures must account for this heterogeneity, and protect at the device level.

Cybersecurity risk assessment

The parallels between safety and cybersecurity are evident in the practice of risk assessment, which is also gaining more attention in cybersecurity contexts. AcuTech's Tom Cottle says "We are now using a HAZOP/PHA [hazard and operability and process hazards analysis] model similar to what you would find for process-safety risk assessment and applying that method to assessing cybersecurity risk."

"Previously, there was a large focus on the severity of a possible incident, where the worst-case scenario was imagined — for example, what if hackers obtained access to the ICS? — but it's hard to say exactly how the severity of an incident would be reduced by the addition of a given cybersecurity measure," Cottle explains. "Now, the focus is more on the likelihood of a cyberincident, rather than solely the severity. We can try to reduce the chances that an attack will be successful, and determine what measures we can take to realistically reduce the likelihood of a successful attack."

As companies approach cybersecurity more holistically, the evaluation of risk improves. "In the past, some risk assessment efforts have had the effect of exaggerating certain risks, while missing others," Cottle says. "Now we are better able to ask questions like, 'How sophisticated would the attacker have to be to have this or that effect?,' and 'How much do they need to know about how our system works to affect it negatively?'"

Of course the "worst-cases" are still very much a concern, but we don't want to focus on only the most potentially destructive and, by doing that, miss something less destructive, but much more likely to occur, Cottle explains.

New offerings

As the sophistication and scope of cyberthreats expand, the methods, strategies and products aimed at cyberdefense are also evolving.

For example, NanoLock's OT Defender product is designed to address the increasing threats and changing security demands. It is a device-level, zero-trust solution that protects the integrity of programmable logic controllers (PLCs) against outside adversaries, supply chain actors and insider incidents, including human errors, the company says, whether they are connected to a network or offline, and whether they are new or legacy systems.

A recent development of the product includes features that respond to market demand. For example: audit trails to provide traceability for operational teams, support for multivendor PLC environments, a failsafe mechanism to ensure business continuity even in times of crisis, and more, NanoLock says.

In addition to growing its Neighborhood Keeper program, Dragos has been constantly updating both its cybersecurity platform and its professional services offerings. "We are continually updating the platform in response to customer requirements," Morris says, and "conducting OT cybersecurity assessments coupled with penetration testing to look for specific vulnerabilities."

White Knight Labs also offers penetration testing services, recently adding a "deep-fake-as-a-service" simulated attack that impersonates voices from recorded audio, as well as continuous threat exposure management, Hatcher says.

In other offerings, Siemens has introduced three software packages that were all developed, tested and used internally at the company before being offered more widely. "We've learned a lot about what works and what doesn't work," says Siemens' Tommey. "For example, Siemens SINEC Security Inspector enables regular and comprehensive checks on the security status of entire OT/IT network environments to identify non-compliance with OT cyber policies and other potential threats at an early stage, which allows planning and scheduling the remediation, the company says. In addition, the company offers Vilocify, a vulnerability intelligence platform for both IT and OT networks, Tommey says.

Meanwhile, the McCrary Institute is developing and offering educational workshops on Cyber-Informed Engineering (CIE). CIE expands cyber "secure-by-design" concepts beyond the digital realm to the engineering of cyber-physical systems, McCrary says.

"CIE uses engineering expertise to evaluate and mitigate cyber risk early in the design stage, using engineering design and controls, not traditional cybersecurity tools," the institute explains, adding that implementing CIE requires "a cultural shift for engineering and cybersecurity teams, and new approaches in research, design, operations, education and standards." ■

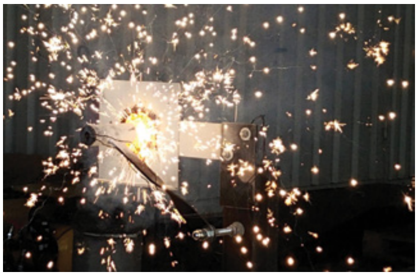
Scott Jenkins

Performance Materials

Lanxess

Remove PFAS from water with this ion-exchange resin

MonoPlus TP 109 (photo) is a new macroporous anion-exchange resin for the selective removal of contaminants, such as per- and polyfluoroalkyl substances (PFAS) from water. Lewatit MonoPlus TP 109 is especially suitable for the purification and remediation of water with PFAS concentrations exceeding 10 parts per billion (ppb). In addition to its high selectivity, the macroporous resin exhibits good kinetics and high fouling resistance, the company says. The uniform bead size ensures improved hydraulics, and the resin can be regenerated with chemicals, such as methanol and sodium chloride. Lewatit MonoPlus TP 109 efficiently binds not only different PFAS, but also complex anions, such as nitrate, bromate, chlorate and perchlorate. It can even remove chlorate from concentrated sodium hydroxide. — *Lanxess AG, Cologne, Germany*
www.lanxess.com



Envalior

This composite passes thermal-runaway tests

Tepex is a new thermoplastic-composite brand that, even with very low-test specimen thicknesses, passes the standard thermal runaway tests (photo) for electric vehicle (EV) battery housings. The high resistance of this composite to the extreme conditions of a battery-cell fire can be attributed to the nonflammable, long and continuous fibers that reinforce the material in a multi-layer structure. The structural material is capable of withstanding the extreme pressures, temperatures well in excess of 1,000°C and bombardment by abrasive hot particles that occur during the thermal runaway of battery cells, the company says. The new composite can pass the standard tests — such as the battery enclosure thermal runaway (BETR) test to UL 2596 — with test specimens having a thicknesses of just 2 mm — or even less, the company says. — *Envalior GmbH, Düsseldorf, Germany*
www.envalior.com

Solvay

A new material for battery thermal-runaway protection

Xencor Xtreme (photo) is a new family of long glass-fiber (LGF) polyphthalamide (PPA) materials for battery applications requiring resistance to thermal runaway and propagation. Xencor XTreme PPA LGF grades are designed to offer superior resistance to direct flame exposure at 1,000°C for over 10 min., providing sufficient time for passengers to exit the vehicle in the event of a thermal runaway, and meeting the latest global regulations in Europe, China, the U.S. and other countries. The materials are designed to retain an excellent level of electrical insulation after exposure to flame, helping to mitigate thermal runaway in batteries. More importantly, the material has a high glass-transition temperature (T_g), which enables dimensional stability of the parts under battery operating conditions. — *Solvay S.A., Brussels, Belgium*
www.solvay.com

AkzoNobel

A coating system saves energy and keeps houses cool

Ceram-A-Star Select Frost coating system (photo) is a tough, durable, two-coat exterior finish using this company's proprietary resins and special additives. The formulation of Ceram-A-Star Select Frost is designed for North American climates. Cool Chemistry pigmentation technology used in Ceram-A-Star Select Frost helps reduce energy consumption by lowering cooling loads in buildings. The standardized palette of Select Frost reduces color complexity and improves operational efficiency. Ceram-A-Star Cool Black coatings are available in both standard and Cool Chemistry options. New blacks provide enhanced options to meet the needs of building construction trends. — *AkzoNobel N.V., Amsterdam, the Netherlands*
www.akzonobel.com

These cable-tie compounds are metal-detectable

This company recently launched precolored, metal-detectable Vydyne PA66 compounds for cable ties and fasteners for industries where foreign-object contamination causes health

concerns and reputational damage, such as in pharmaceutical and food-processing facilities. The new product meets the most stringent performance standards and simplifies the supply chain for cable tie and fastener producers. The new metal-detectable Vydyne reduces the need for additional tolling or masterbatch steps and ensures the material performs as intended. The company ensures the consistent dispersion of the metal additive within the polymer matrix, so that even small metal particles are clearly visible and identified with standard detection equipment. — *Ascend Performance Materials Operations LLC, Houston*
www.ascendmaterials.com

This powder-coating texturing agent is PTFE-free

Ceridust 8170M (photo) is a polytetrafluoroethylene (PTFE)-free alternative texturing agent for powder coatings. The development addresses the concerns about PTFE's impact on human health and the environment, which is leading to the potential restriction of its use. Ceridust 8170 M combines the texturing qualities of PTFE and the environmental benefits of a PTFE-free alternative. Ceridust 8170 M also has the added benefit of reduced energy consumption as part of the powder-coating extrusion process. Also, Ceridust 8170 M is compliant with the E.U. REACH regulation and is not only PTFE-free, but also PFAS-free (per- and polyfluoroalkyl substances). — *Clariant International Ltd., Muttenz, Switzerland*
www.clariant.com

Protect workers from hot surfaces with this barrier

Belzona 5871 is a two-component, polymeric, solvent-free system providing a thermal insulation barrier with corrosion protection and thermal and sub-zero "cool-to-touch" properties. This material is designed to be applied to metal pipework, ducting and other industrial equipment. In a recent application, the barrier was applied to the upper surface of a tank (photo) that reaches temperatures of 110°C, to protect employees from the risk of contact burns. Belzona 5871 can be applied by brush, cartridge or plural spray, expanding upon application to produce a lightweight, closed-cell foam. This reduces the surface temperature of metallic substrates to below 60°C, while providing corrosion protection. — *Belzona Polymers Ltd., Harrogate, U.K.*
www.belzona.com

A polymer formulation for roofing weathering protecting

Luran S is an ASA (acrylonitrile styrene acrylate) polymer that offers long-term performance when exposed to ultraviolet (UV) light and heat. Its low maintenance and high weather resistance make Luran S suitable for a wide range of outdoor applications. Mixing with polyvinylchloride (PVC) or covering over PVC makes Luran S suited for applications such as roof tiles, gutters or drain down pipes. Luran S ECO is this company's first sustainable ASA solution with up to 39% lower carbon footprint. EdilPlast SRL recently selected this material for Cover Innovation, a new roofing sheet product (photo). The use of these materials will allow EdilPlast to use from 30% to 50% bio-attributed raw materials. — *Ineos Styrolution Group GmbH, Frankfurt am Main, Germany*
www.ineos-styrolution.com ■

Gerald Ondrey

Beckoff Automation

Pfeiffer Vacuum

Emerson

HIMA Paul Hildebrandt

Gericke USA

Water-cooled servomotors for extreme dynamic requirements

The AM8300 servomotors expand this company's portfolio to include a modular motor series with integrated water cooling (photo). Due to the efficient cooling, an extremely high power density is achieved, so that a rated power of up to 40 kW can be delivered in the smallest installation space, depending on the size. Compared to similar conventional convection-cooled motors, the standstill torque is three times higher, the company says. The servomotors offer maximum dynamics, because the torque increases with water cooling, but the rotor moment of inertia remains constant. These are particularly suitable for applications with higher speed and torque requirements. — *Beckhoff Automation GmbH & Co. KG, Verl, Germany*
www.beckhoff.com

Residue-gas analyzers for cleanliness verification

OmniGrade (photo) is a residual-gas analysis (RGA) system that is designed to meet specific testing demands. OmniGrade configurations include two or three chambers: the spectrometer chamber in which the mass spectrometer is positioned, and a measurement chamber where the samples are placed during measurement. Options include a load lock chamber to minimize system background and an automated sample transport system. Cleanroom compatibility is possible. The mass spectrometer can be selected according to the application (PrismaPro or HiQuad). Thermal heating, either of the pure system or together with the sample (bake-out), will further optimize the measuring capability and improve sample cleanliness. — *Pfeiffer Vacuum GmbH, Asslar, Germany*
www.pfeiffer-vacuum.com

These solenoid valves handle high flow with minimum power

The new ASCO Series 327C solenoid valve (photo) features a direct-acting, high-flow design that provides superior flow-to-power ratio compared to similar valves. The Series 327C design features a balanced poppet construction that permits high flows at minimum power levels, making it suitable for use in power plants, petroleum refineries and chemical-processing facilities. The valve features a unique, two-layer dynamic-seal technology that provides low friction and excellent stiction resistance, helping ensure reliable valve operation in environments with temperatures ranging from –60 to 90°C. In addition, the valve is SIL (safety integrity level)-3 capable, demonstrating a high level of performance integrity and a very low risk of failure over the valve's projected lifecycle. — *Emerson, St. Louis, Mo.*
www.emerson.com

A hub for the digitalization of data from safety devices

Whether in the engineering of large and distributed automation projects, in the diagnosis of faults or in regular testing, the specification, configuration and maintenance of safety devices in process plants requires effort and careful attention. If new functions are added to components of safety devices, time-consuming recertification was traditionally necessary. The next version of SILworX (photo) helps to reduce this effort through consistent digitalization and new functions in order to simplify these tasks. A distinction is made between core functions for programming safety controllers and Industry 4.0 functions. New functions that are not safety-related can be integrated using modular plug-ins via an interface that does not affect the core safety-rated SILworX functions. This modular structure makes it possible to digitalize automation processes quickly and flexibly. — *HIMA Paul Hildebrandt GmbH, Brühl, Germany*
www.hima.com

Bulk material feeder ensures consistent product flow

The Feedos S bulk material feeder (photo) automatically meters and doses powders and other dry materials with the high accuracy and consistency needed to optimize continuous processes. Featuring a proprietary design, the Feedos S is engineered with optimized geometry that eliminates dead zones to promote smooth, uninterrupted flow of a wide range of free-flowing and non-

flowing materials. Suitable for food, dairy, chemical, pharmaceutical and other manufacturers, the Feedos S ensures a non-stop flow of material to mixers, blenders, compounders, reactors and other continuous processing equipment at rates up to 500 L/h. Additional models achieve throughput rates up to 54,000 L/h. — *Gericke USA, Inc., Somerset, N.J.*
www.gerickegroup.com

A workstation with an aluminum housing for harsh conditions

This company expanded its portfolio of VisuNet FLX series operator and monitoring workstations (photo) with a version for outdoor use and use in an extended temperature range from –20 to 50°C. The new aluminum housing not only ensures an optimal heat sink, but also offers exceptional resistance to more demanding outdoor conditions. The low weight ensures easy installation via a wide range of mounting options. The VisuNet FLX series has an optically bonded display to ensure perfect readability in direct sunlight. In addition, various shading elements can be selected as required. The integration of an optional RFID reader simplifies user authentication and contributes to security in industrial environments. The series is available as a complete human-machine interface (HMI) system with aluminum or stainless-steel housing, as a panel mounting or as a box PC, each with display sizes of 15.6, 19 or 21.5 in. — *Pepperl+Fuchs SE, Mannheim, Germany*
www.pepperl-fuchs.com

Digital service significantly reduces compressed-air costs

The Monitoring Box FTMg Premium (photo) is a new digital service for compressed-air monitoring. In addition to continuous monitoring, the Cloud solution from this company's Field Analytics Portfolio can identify consumption losses due to machine- or process-related inefficiencies and detect leaks at an early stage, reporting them with an alarm. Moreover, the digital service can compare sources of compressed-air consumption on the basis of their consumption and attendant costs and suggest optimization options. The benefits include reduced production costs, a smaller CO_2 footprint and more-efficient service planning. With the Monitoring Box FTMg Premium, production planners, energy managers and maintenance engineers can ultimately reduce their company's compressed-air costs by up to 30%, the company says. — *SICK AG, Waldkirch im Breisgau, Germany*
www.sick.com

Optimized dosing solution for drip applications

The newly developed vipro-Pump MR series (photo) offers plant operators a virtually maintenance-free dispenser that doses all conventional impregnating resins — even chemically aggressive ones. A patent application has been filed for the vipro-Pump MR and the associated process. The dosing system features an adapted seal and pump housing with material reservoir; a freely "teachable" fill level sensor for flexible adaptation to the production process; temperature monitoring; and more. The optimized dispenser is available in two sizes: vipro-Pump 40 MR and vipro-Pump 100 MR. These sizes cover the typical process speeds for applications such as impregnating motor stators. — *ViscoTec Pumpen- u. Dosiertechnik GmbH, Töging a. Inn, Germany*
www.viscotec.de

Integrated sifter and bag-break station prevents contamination

This company has custom-engineered an integrated sifter and bag-break station that automatically removes foreign matter and captures nuisance dust. Set above a top-loading batch mixer, the powder-handling system features an AFC Dump Clean bag-break station (photo) that continuously draws any fine particles inside the unit before they can escape into the workplace and a vibratory sifter that separates and diverts any "tramp" material from the product stream. When discharged for mixing, the product is protected from contamination, and the mixer and other downstream equipment are protected from tramp metals, stray bulk-bag material and other unwelcome waste. — *Automated Flexible Conveyor, Inc., Clifton, N.J.*
www.afcspiralfeeder.com ■

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Automated Flexible Conveyor

Metal-Organic Frameworks: Industrial Applications

Department Editor: Scott Jenkins

Metal-organic framework materials (MOFs) have garnered considerable attention as candidate materials for a number of applications, such as gas storage, molecular separation and catalysis, because their organic and inorganic components are highly tunable at the molecular level. Since being introduced in the 1990s, more than 100,000 types of MOFs have been reported [*1*]. However, only a few MOFs have been synthesized on a large scale [*2*]. This one-page reference outlines the challenges of commercial production and the application areas where MOFs are being developed for industrial use.

MOF properties

MOFs are porous crystalline macromolecules formed by the coordination of cation-containing metal nodes called secondary building units (SBUs) — formed by the coordination of metal ions and other atoms — with organic ligands (Figure 1). The combination results in a controllable, three-dimensional network structure at the nanoscale, featuring highly ordered pore structures with extremely high surface areas (1,000–10,000 m^2/g). Because different organic ligands (such as di- and tricarboxylic acids) and metals (such as zinc, zirconium, aluminum, iron, copper and others) can be used, MOFs have broad structural diversity, allowing them to be produced with desired porosity and with added functionality, such as hydrophilicity or hydrophobicity.

MOF commercial production

Although researchers have produced a large number of promising MOFs at the gram scale using an array of synthetic techniques, large-scale production of MOFs presents significant challenges. The market for MOFs is expanding, but remains limited, which restricts large-scale production. Further, other factors related to raw materials, process feasibility and cost are also at play. The lack of stability and reproducibility, the use of expensive organic linkers, the use of metal precursors that are corrosive, or the use of toxic and expensive solvents, still hamper large-scale MOF production.

Companies that have successfully produced MOFs at large scale include BASF SE (Ludwidshafen, Germany; www.basf.de), Framergy Inc. (College Station, Tex.; www.framergy.com), Nuada (Belfast, U.K.; www.nuadaco2.com), ProfMOF AS (Kongsberg, Norway; profmof.com), Promethean Particles Ltd. (Nottingham, U.K.; www.prometheanparticles.com) and novoMOF AG (Zofingen, Switzerland; www.novomof.com) [*2*].

Application areas

The following are the main areas of industrial application for which MOFs are being developed [*3–5*]:

Gas storage. The characteristic high surface area of MOFs aids in adsorption of gases, while pore volume determines the amount of gas that can be stored in the structure. Most MOF development focuses on methane, H_2 and CO_2 storage due to their importance in energy sectors.

Gas adsorption and separation. MOFs can be used for selective adsorption and separation of gases from a mixture. Selective adsorption is attractive in the many practical applications where gases are mixtures.

Catalysis. Interest in MOFs as catalysts is driven by their ability to offer a high density and uniform dispersion of active sites. Catalytic properties of MOFs may result directly from their hybrid structure or can be the result of incorporated catalytically active nanoparticles. Pores and channels of the MOF framework can facilitate the accessibility of active sites and the transport of substrates and products. Tailoring pore sizes and shapes may allow MOFs to perform as shape-selective catalysts.

Energy storage. MOFs also show great potential for rechargeable batteries and supercapacitors. MOFs are being explored for electrode materials for lithium-ion, sodium-ion and lithium-sulfur batteries. The concept

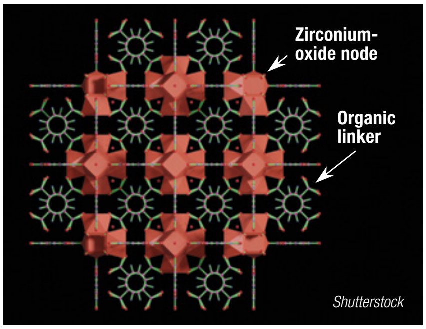

FIGURE 1. In an example showing MOF structure, the MOF known as UiO-66 contains Zr-based nodes and benzenecarboxylic-acid-based linkers

is to use the high surface area and permanent pores of MOF structures to allow efficient Li^+ ion storage and migration during charge and discharge cycles.

Wastewater treatment The tunable porous structure of MOFs allows the selective separation of organic and inorganic substances from water.

Sensors. Tunable structure and porosity make MOFs and their composites attractive as chemical sensors. One class of MOFs exhibits luminescent behavior upon the inclusion of a guest molecule. This unique function is useful for sensor applications.

Hydrogen generation. MOFs may be utilized for hydrogen production via photocatalytic and electrocatalytic production processes.

Biomedicine. The structural diversity of MOFs, along with high surface area and large pores could make them a target platform for biomedical applications. They are being explored as novel drug-delivery systems and for the controlled release of compounds. Some MOFs show biodegradability and high loading capacity in biomedical applications. ■

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Corrosion Prevention for Heating & Cooling Systems

Understanding that viable strategies exist to protect essential cooling and heating systems from corrosion is a crucial step in ensuring proper asset protection

Scott Bryan and Julie Holmquist
Cortec Corp.

IN BRIEF

HEATING AND COOLING IN THE CPI

SEASONAL ADJUSTMENTS

CORROSION OF BACKUP EQUIPMENT

DIFFERENT RISKS FOR DIFFERENT METALS

COSTS OF A CORRODED SYSTEM

COMMON METHODS OF PROTECTION

A PROACTIVE APPROACH

SEASONAL DRY LAYUP OF BOILERS

SEASONAL LAYUP OF COOLING UNITS

Several years ago, a manufacturing plant was forced to unexpectedly shut down one of its main production lines for several weeks. The cause: a rusty vacuum pump that pulled water through the cooling loop had corroded to the point of failure. Production on that line shut down for several weeks until a new pump could be delivered, costing the company not only the price of the new pump, but also the earnings that would normally have been generated if the manufacturing line had been in use. Perhaps the most troubling part of the problem was that the pump failure and line shutdown would have been completely avoidable had the manufacturer been using corrosion inhibitors in its cooling water. Unfortunately, the maintenance team was apparently unaware that this was even an option. With the discovery of the problem, the plant adopted a new water-treatment plan for the closed-loop system going forward, and also applied it to the company's two other chiller systems [*1*].

The story above underscores the importance of engineers, facility managers and maintenance teams knowing their options when it comes to the proper upkeep of their process heating and cooling systems. Cooling and heating are integral parts of the chemical process industries (CPI), and without them, many processes simply cannot function. While one would hope that the natural ingenuity and problem-solving skills of mechanically-minded maintenance teams would know all the tricks of the trade to keep a chemical plant functioning optimally, this should not be taken for granted. In every industry, professionals must continue to learn and grow in their skills while staying abreast of technologies that can improve routine maintenance. One of these areas is corrosion protection, which can have a significant impact on the longevity of process heating and cooling equipment and whether or not unwelcome corrosion-related interruptions occur. Awareness of the proper tools for prevention can make a significant difference.

Heating and cooling in the CPI

Heating and cooling play a variety of roles within the CPI. Mixing tanks where chemical reactions occur often need to be cooled down with a jacket of cooling water. In other cases, raw materials need to be heated up prior to mixing. For this purpose, many CPI plants have boilers that supply steam throughout the plant to appropriate points where heating is needed. Sometimes, chemicals that are manufactured must be stored at extremely cool temperatures and only heated up when they are ready to be applied. Fractionation towers at petroleum refineries are one specific example of a chemical process that relies on chillers, heat exchangers and reboilers to convert hydrocarbons into various products. The list goes on to encompass many other examples, including boilers and cooling systems used as part of heating, ventilation and air conditioning (HVAC) systems for comfort heating and cooling.

FIGURE 1. This compressor-based chiller does not operate in temperatures below 55°F. While the inside of the heat exchanger (cylinder at right) is made of copper and therefore is less likely to corrode, the outer shell is likely made of carbon steel and could therefore especially benefit from exposure to corrosion inhibitors during the off-season when coolant is sitting stagnant in the system

Seasonal adjustments

While many process heating and cooling systems remain in operation year-round, there are some seasonal changes that affect individual components or require seasonal layup of HVAC systems. One example is when plants switch between compressor chillers and air-cooled systems to regulate the temperature of their closed-loop coolants. Compressors located outside in regions with cold winters may not be able to operate when temperatures drop below 55°F for long periods of time (Figure 1). At this point, the plant may transfer over to an air-cooled system that takes advantage of cold or freezing outdoor temperatures to cool off the hot glycol passing through the chiller before it heads back into the building. This may mean switching back and forth seasonally between the pump that sends the coolant to the compressor-powered chiller versus the pump that sends it to the air-cooled chiller (Figure 2). As a result, one or more pumps or heat exchangers remain offline, either partially filled with stagnant coolant or empty altogether. This is a prime time for corrosion to occur if the cooling water is not properly treated. Seasonal facility heating or cooling changes also heighten corrosion risks for boilers or cooling towers that go offline for months at a time. It is often during these times of seasonal layup that corrosion strikes, because the normal operating chemical program is not in circulation.

Corrosion of backup equipment

A similar story can be true for backup equipment. Redundant pumps, meant to ensure that coolants or hot water keep flowing through the system if the primary pump fails, are more vulnerable to corrosion when temporarily offline — even if plants frequently alternate between pumps to reduce the risk. Unfortunately, a backup pump that is corroded and does not work properly can be as bad as not having a backup at all.

Another common piece of backup equipment is the steam-producing boiler kept on standby. This setup eliminates the need to refill or completely reheat the boiler water to keep the steam supply going if the primary boiler breaks down or has to be taken offline for routine servicing. While this backup plan minimizes interruptions to plant operations, typical methods of wet boiler layup require a great deal of maintenance work and may therefore go by the wayside. These methods also do not typically address corrosion concerns in the headspace above the boiler water where moisture may condense.

FIGURE 2. This cooling loop uses multiple pumps. When the temperature drops below 55°F, the left pump kicks in to pump glycol to the air-cooled chiller. When the temperature rises above 55°F, the right pump turns on to pump coolant to the compressor-powered chiller. Pump internals are often made of carbon steel or cast iron, making them good candidates for corrosion protection, especially during offline periods

Different risks for different metals

It is crucial that engineers understand that different types of metal have varying levels of corrosion risk. This is an important consideration for engineers designing the system, who must weigh the benefits of using a more expensive corrosion-resistant alloy, such as titanium or copper, against staying within budget by using cheaper but less corrosion-resistant metals, such as carbon steel and cast iron. Pumps and heat-exchanger shells are frequently made with carbon steel, while piping and internal heat-exchanger tubing are often made with copper or other more thermally conductive alloys. Once the system is designed and installed, maintenance personnel must be prepared to pay special attention to components made of metals susceptible to corrosion, although even yellow metals (brass alloys) can be at risk.

Costs of a corroded system

Vulnerabilities aside, what are the costs of corrosion in heating or cooling systems? The answers are many and varied. One common effect is that corrosion byproducts will migrate throughout the system, causing both fouling and the potential for even more corrosion sites to form. Furthermore, any metal loss from corrosion means a potentially shorter service life for the equipment. The buildup of corrosion products on heat-exchange equipment can significantly reduce a unit's designed heat-transfer efficiency. If the corrosion is severe enough, it can cause equipment failure, opening up an entirely new set of problems. These new problems may include the cost of buying a replacement asset, the cost of labor needed for removal and installation and the cost of production lost while the equipment is out of operation. The bigger the operation, the higher the stakes, so prevention is almost always the more economical option.

Common methods of protection

Some common corrosion inhibitors for cooling loops include sodium nitrite, sodium molybdate and polymeric formulations [*2, 3*]. Among other things, one of the drawbacks of these treatments is that they do not provide corrosion protection in the vapor phase, neglecting protection for any area not in direct contact with the treated coolant (for example, pumps or systems that are offline and drained, or the headspace in pipes and systems that are partially filled). In the case of boiler layup, the use of quick lime or desiccant is common, with the main goal of absorbing as much moisture as possible. Boilers on wet layup or standby are often protected by

keeping an elevated pH and higher sulfite residuals, an approach that requires frequent monitoring and does not address corrosion above the water level. Sometimes, nothing is done at all, accelerating the path to corrosion, downtime and shorter service life.

A proactive approach

Another potentially more proactive approach is to use vapor-phase corrosion inhibitors, such as amine carboxylates, which work in both wet and dry systems. In addition to dissolving in the system fluid and protecting any metals in direct contact with it, these corrosion inhibitors also have the ability to vaporize and diffuse throughout small air pockets or large void spaces. This trait has obvious advantages for an empty backup pump, a cooling system that has been drained for the season or the headspace in a boiler on standby. These corrosion inhibitors, whether in vapor or liquid phase, form a protective molecular layer on the metal surface, interrupting the normal electrochemical reaction that takes place in the presence of metal, oxygen and an electrolyte. Protection is usually mixed, inhibiting a corrosion reaction at both the anode and cathode of a potential corrosion cell. Corrosion prevention with vapor-phase inhibitors takes many forms depending on the component and the stage of use. Some hypothetical examples follow.

Closed loops and pumps. Closed-loop cooling systems should be regularly treated with corrosion inhibitors as part of the normal chemical operating program (Figure 3). This reduces the risk for problems similar to those experienced by the manufacturer with the corroded vacuum pump mentioned at the opening of this article. Routine treatment with vapor-phase corrosion inhibitors also means that the system is prepared for times when, for example, the pump to the compressor chiller is turned off to allow the air-cooled chiller to take over. Protection will continue on metals in stagnant areas, as well as those that are not completely filled. With the right corrosion inhibitor selection, protection can also cover multiple alloys, including carbon steel, cast iron and copper, at once.

Boilers and steam systems. Redundant boilers kept on low fire can sometimes be protected with the same package of corrosion inhibitors used for closed-loop systems if the temperature is not extremely high. Compared to a traditional sulfite program, application of vapor-phase inhibitors is typically much safer and more convenient. The vapor-phase action takes care of protection above the water level, in addition to the protection that is going on below the water level (Figure 4). Frequent monitoring is not required, and the system can be turned back on at any time to continue necessary operations.

FIGURE 3. Chemical reactions in this tank give off heat, requiring coolant to be piped into the reactor to cool the process down. Vapor-phase corrosion inhibitors are added to the coolant on a regular basis for corrosion protection of metals inside the closed loop

Seasonal dry layup of boilers

Boilers used for facility heating are often shut down and drained completely, constituting a dry boiler layup. The use of vapor-phase inhibitors at this stage is a major improvement on desiccant-only layups, which seek to minimize the source of the problem by absorbing moisture but not proactively fighting corrosion. If the desiccant is unable to absorb all the moisture present, corrosion may occur in spite of protective efforts. For extended shutdowns of 30 days or more, the desiccant should be monitored and replaced as necessary to maintain a moisture-free system. Workers must also be sure to

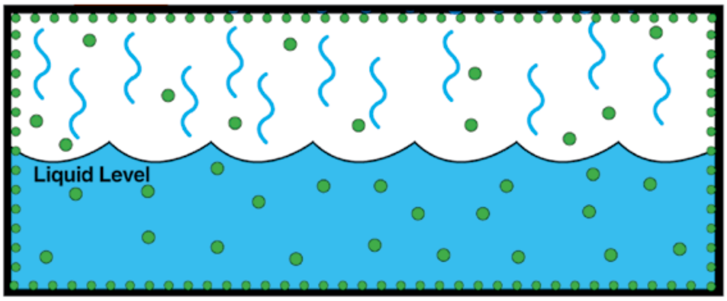

FIGURE 4. Amine-carboxylate based vapor-phase corrosion inhibitors protect below and above the fluid level, forming a protective molecular layer on metal surfaces

remove the desiccant before putting the boiler into operation, or further complications will develop.

In contrast, protection with vapor-phase inhibitors is convenient, comprehensive and proactive. It can be as simple as placing a water-soluble pouch of vapor-phase inhibitors inside an empty boiler and slitting it open to allow the release of corrosion inhibiting vapors. As long as the boiler remains closed, these corrosion inhibitors actively protect all accessible metal surfaces within the boiler. One advantage is that the boiler does not have to be completely dry in order for the corrosion inhibitors to work. Another advantage is that bringing the boiler back online is as easy as filling the boiler back up since the corrosion inhibitor pouches simply dissolve in the boiler feedwater. For extremely large boilers, vapor-phase inhibitors are available as a waterborne fluid that can be fogged throughout the boiler and steam system, typically without requiring removal before startup.

Seasonal layup of cooling units

Corrosion-inhibitor application for seasonal shutdown of cooling systems is usually slightly different. It often consists of adding the corrosion inhibitor to the system, circulating it and letting it stand for 24 h to allow the formation of a protective film on the cooling system walls. The loop can be left full for wet layup or drained for a dry layup. In either case, vapor-phase corrosion inhibitors remain to help protect all accessible internal areas of the system, provided the system openings are closed. Often, the alternative is either to do nothing or to hope that loading the system with corrosion inhibitors from the operational water-treatment program will provide protection even after the system has been drained — which is typically a fruitless effort unless amine-carboxylate-based corrosion inhibitors have been used.

The benefits of such corrosion avoidance and asset protection in the CPI are far-ranging. From reducing the high cost of downtime and sidestepping the complications of rust clogging to extending equipment service life and improving heat-exchanger efficiency, these benefits begin with simple awareness of the prevention measures that are available. ■

Edited by Mary Page Bailey

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All images courtesy of Cortec.

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Authors

Julie Holmquist (Email: jholmquist@cortecvci.com) has been a content writer at Cortec Corp. since 2015. She specializes in writing about corrosion-inhibiting technology for a variety of industries, including manufacturing, oil and gas, power generation and water treatment.

Scott Bryan (Email: sbryan@cortecvci.com) is Cortec's technical sales manager for water treatment products. He has almost 30 years of experience in the water treatment industry. Bryan has maintained his qualifications as a Certified Water Technologist (CWT) since 2005. For 15 years, Bryan worked as a distributor for Cortec water-treatment solutions before joining Cortec in 2019.

From Waste to Resource: Avoiding Fouling in Ammonia Recovery

As more organizations look to ammonia recovery to take advantage of recent pricing trends, it is increasingly important to consider the impacts of fouling and other process inefficiencies

Chris Rentsch and Allan Fox
Koch Modular Process Systems

The price of ammonia has increased by 600% from 2020 to 2022 (Figure 1) [*1*]. In turn, a wide range of ammonia recovery projects that were previously not economical have become attractive. Ammonia recovery technology converts waste ammonia into a useful resource, but fouling can be a significant issue in these processes. Fouling occurs when unwanted substances accumulate on heat-transfer surfaces, reducing the efficiency of the ammonia recovery process. Effective fouling-control strategies are therefore essential to ensure efficient and sustainable ammonia recovery operations.

Common fouling-control strategies include chemical cleaning, mechanical cleaning and filtration. Implementing these strategies early in the design process is important to minimize fouling and maximize ammonia recovery efficiency. Furthermore, an understanding of the different equipment and process-technology configurations during the early design stage can help to reduce the risks for fouling. This article examines various ammonia recovery technologies and discusses design considerations related to fouling.

Ammonia recovery technologies

An engineer may be tasked with recovering ammonia from aqueous streams of varying purity, composition and flowrate. The presence of particulate solids and other soluble species, as well as the process pH, also present a challenge. To ensure successful ammonia recovery, the engineer must select the appropriate ammonia recovery technology based on the specific characteristics of the aqueous streams. Ammonia may exist in solution in free or fixed forms. Free ammonia readily dissociates upon heating (for example, $NH_4OH \leftrightarrow NH_3 + H_2O$) and is readily liberated as a vapor. Fixed ammonia (such as NH_4Cl) requires adjusting the pH into the alkaline range, converting nonvolatile ionized ammonia into volatile free ammonia.

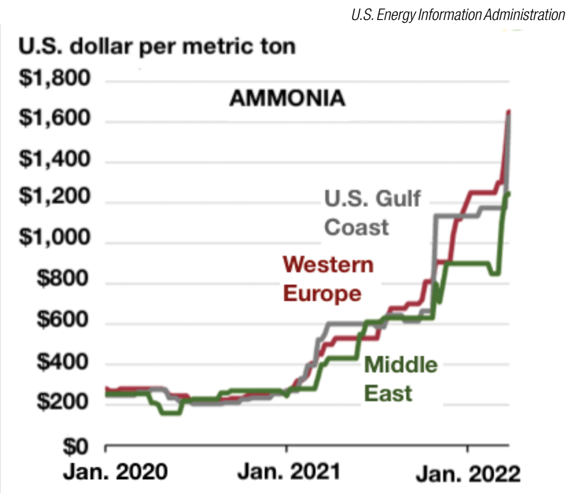


FIGURE 1. As ammonia prices have significantly risen in recent years, many processors are investing in ammonia recovery technologies [*1*]

There are several technologies commonly used for ammonia recovery including the following, each with its own benefits and challenges in terms of process efficiency and fouling propensity:

- Distillation (stripping) towers
- Dilute acid neutralization and crystallization
- Membrane or reverse osmosis separation

Engineers must carefully consider which technology is best suited for their specific application. Distillation is one of the most common technologies used for ammonia recovery, owing to the relatively low capital cost, ease of use, proven technology and operational reliability. Aqueous ammonia-bearing feed material flows down through trays or packing, while steam injected at the lowest point vaporizes the ammonia and carries it out as distillate. Distillation requires no ongoing consumption of chemicals or high pressures, in contrast to crystallizer systems that consume stoichiometric quantities of acid (typically sulfuric acid) or require application of high pressure to force water through a membrane.

Ammonia stripping challenges

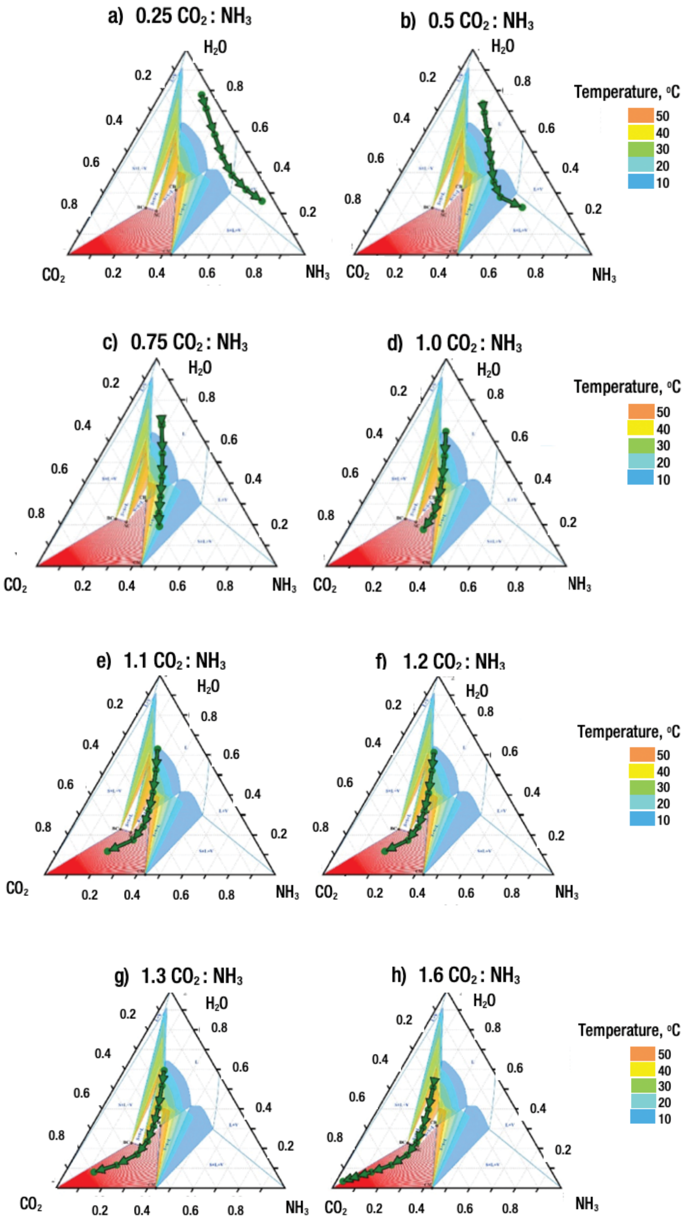
Quite commonly there are more species than just water and dilute ammonia present in the feed liquid. Aqueous ammonia generated during biomass processing or fermentation often is mixed with dissolved carbon dioxide, in the form of carbonic acid. Carbonic acid is acidic, while

ammonia is alkaline. One serves as the neutralizing agent for the other. Two salt combinations typically result: ammonium carbonate or ammonium carbamate. Steam stripping of NH_3 will also strip the CO_2 out of the water, and if not correctly designed, the two gas species will recombine in the stripper overhead condenser, leading to fouling with salt deposits. This will eventually reduce performance to below the design production rate. This is because there is a composition gradient from the inlet to the outlet of the condenser. While a simulator may show a completely dissolved solution of ammonium carbonate at the condenser outlet, one must be aware that the condenser feed vapor does not instantly condense and may pass through insoluble regions.

One helpful way to visualize this is to use thermodynamic modeling software that includes a properties package for sour water to calculate equilibrium constants for ammonia and carbon dioxide in water. The portion of the incoming vapor that condenses incrementally increases down the length of the condenser. As 10%, then 20%, and so on up to 100%, of the feed vapor condenses, the residual vapor-phase composition can then be overlaid onto a ternary phase diagram, such as that developed by Sutter and others, shown in Figure 2 [*2*]. The dark green arrows originate at the inlet composition of the initial vapor phase and progress toward the final residual vapor composition occurring just before the vapor is entirely condensed. The procedure is repeated for many concentrations of CO_2, expressed as mass ratios to the ammonia. All pathways shown are based on an initial ammonia composition of 17.5 wt.%. At different initial compositions, the thresholds discussed below may be different and need to be reevaluated.

When the mass ratio of CO_2:NH_3 is low (≤0.25, Figure 2a) the entire green condensing curve, or "pathway" across the ternary diagram, is in regions of liquid and vapor phases only. A standard shell-and-tube condenser is expected to perform satisfactorily in this scenario. When the ratio of CO_2:NH_3 increases to 0.50 (Figure 2b), the initial and final compositions of the pathway are liquid and vapor, but the intermediate passes through a region containing a partial solid phase containing ammonium carbonate and ammonium carbamate at cold temperatures (10°C). A shell-and-tube condenser is expected to perform satisfactorily, provided the wintertime cooling water temperatures stay warmer than 20°C. If colder cooling-water temperatures cannot be avoided, a shell-and-tube condenser may suffer fouling. This would be further exacerbated at higher ratios of CO_2:NH_3 (0.75; Figure 2c) where the minimum temperature to avoid the formation of a solid phase is now around 40°C. A shell-and-tube condenser is virtually certain to suffer fouling unless a tempered loop is utilized to keep the cold utility at around 50°C.

When the ratio of CO_2:NH_3 is high (≥1.0, Figure 2d and 2e), the final condensing vapor composition is in a region where fouling is expected to be the most severe and will form a variety of ammonium salts. The solid fouling layer is expected to decrease heat transfer rates and force a tower rate reduction. A condenser wash-out would be necessary to (temporarily) restore production rates.



Ternary diagram courtesy of Sutterand others. Used with permission

FIGURE 2. Ternary diagrams show the compositional curve pathways in overhead condensers of ammonia stripper towers with various CO_2:NH_3 ratios. Initial conditions for all curves are based on 17.5 wt.% ammonia [*2*]

DCC benefits

The design solution for higher CO_2:NH_3 ratios (≥0.75) is to replace the overhead shell-and-tube condenser with a direct contact condenser (DCC; Figure 3). Rather than presenting a metal heat-transfer surface, the DCC sprays a subcooled liquid distillate onto the vapor stream exiting the ammonia stripping tower. The latent heat of vaporization is absorbed as sensible heat into the liquid spray, condensing the vapor. The heated liquid is then cooled via heat exchange in an external recirculation loop before returning to the DCC spray nozzle. A slip-stream is

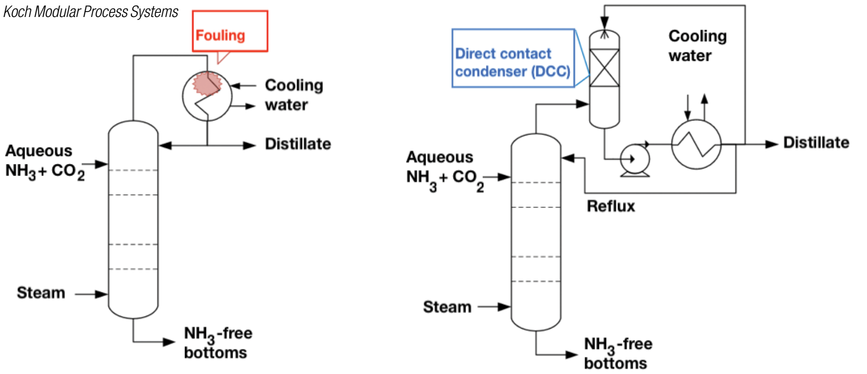


FIGURE 3. A standard ammonia stripper with a shell-and-tube condenser is shown on the left. On the right is an ammonia stripper layout that includes a direct contact condenser (DCC)

withdrawn as product or reflux to ensure the overhead liquid inventory remains constant over time.

The primary advantage conferred by the DCC is compositional control — the mass flowrate of circulated liquid as a ratio to incoming vapor is >10:1 to ensure that there is enough sensible heat to fully condense the incoming stream (except for any non-condensables). Therefore, even small initial amounts of vapor condensation do not materially change the composition of the liquid phase, as it is impossible to enrich the condensed phase in CO_2 and NH_3 when most of the mass comes from the liquid spray, not the vapor feed. A DCC also virtually eliminates dry spots on the condenser, which are prone to solid buildup. Any solids that do form would either be washed away or dissolved in the water-rich wash stream. Viewed on a ternary diagram, the DCC effectively operates on a single point, rather than a pathway, and that point is the initial point of the pathway for the respective CO_2:NH_3 ratios. The initial point existing in the region of liquid and vapor only avoids solids formation, which would foul or plug the system. Therefore, a DCC-equipped ammonia stripper is tolerant to a much wider range of CO_2 concentrations in the stripper feed than a shell-and-tube condenser.

When the ratio of CO_2:NH_3 is even higher (≥1.2, Fig. 2f, 2g and 2h), even the initial composition of the condenser (or DCC) would be prone to fouling if it were to reach a low enough temperature, based on the given ratio. If, during the winter months, the cooling water were to cool the recirculating stream into solid forming regimes, fouling could even form in the DCC and the recirculation cooler. To solve this issue, a tempered loop should be added on the recirculation cooler of the DCC to maintain a temperature above 50°C. When operating at atmospheric pressure, the bubble point temperature of 17.5 wt.% ammonia is around 71°C. If the CO_2:NH_3 ratio is such that the initial DCC composition is already prone to forming solids, this will require a very large recirculation flow in the DCC, in order to straddle the line between fully condensing the vapor stream and not forming solids in the DCC and recirculation cooler. This would lead to much larger operating equipment. In these circumstances, it is advisable to operate the stripping tower at a higher operating pressure. This will increase the bubble point temperature of the ammonia distillate to give more allowance for a temperature rise in the recirculation fluid, while reducing the required recirculation rate. Unfortunately, increasing the operating pressure also has some downsides since it will lead to increased capital cost and require more steam to strip the same amount of ammonia.

The capital cost for a DCC is higher, owing to the higher equipment footprint and the larger heat exchanger size. The process temperature is dependent on operating pressure and typically subcooling the recirculated fluid by about 50°F is sufficient. A DCC heat exchanger will have a larger surface area to remove the same amount of heat as a conventional overhead condenser, owing to the lower temperature differential between the process and utility fluids.

The DCC resembles a miniature stripping tower, often with tower packing inside a cylindrical shell, albeit much shorter than the stripping-tower packed section. The packing ensures sufficient mass-transfer area between the hot condensing vapor and the cooled incoming recirculation liquid. Special consideration must be given to the packing type, particularly in applications with higher liquid loading or rapidly decreasing vapor volume.

It is impossible to avoid co-stripping the CO_2 and NH_3 simultaneously if the ammonia must be recovered. To separate CO_2 from NH_3, one must utilize sequential stripping towers — a CO_2 stripper followed by an NH_3 stripper. Whether one or two stripping columns are required is dictated by the product specifications the system must achieve. Ultimately, the engineer must carefully evaluate the specific characteristics of their process streams and consider all available options to determine the most appropriate ammonia recovery technology for their application. ■

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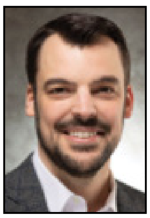

Chris Rentsch is the process development manager at Koch Modular Process Systems (45 Eisenhower Drive, Suite 350 Paramus, NJ 07652; Email: contact@koch-modular.com; Phone: 877-780-8654). He has spent the last 20 years in various production and process engineering roles in many industry sectors, including oil-and-gas, herbicides, lithium-ion batteries, and biomass-to-renewable chemicals. Rentsch holds a B.S.Ch.E. from the University of Michigan.

Allan Fox is a senior process engineer at Koch Modular Process Systems (same contact information as above). Fox has spent the last five years in process engineering roles, and the last 13 years in heat-exchanger design for various industries. He holds a B.S.Ch.E from the State University of New York at Buffalo.

Steam Reboilers: Condensate Vessel Balance to Reboiler is Important

Properly locating a simple 1–2-in. balance line can make all the difference in successful reboiler operation

James R. Risko
Consultant (retired president, TLV Corp.)

IN BRIEF

ISSUES AND THEIR CAUSES

USING THE EXTENDED STALL CHART

VAPOR BALANCING BASICS

BALANCING OPTIONS

DE-ETHANIZER REBOILER: CASE REVIEW

DE-ISOBUTANIZER REBOILER: CASE REVIEW

Steam reboilers are crucial to production in petroleum refineries and petrochemical plants, representing huge capital investment in distillation columns. Regardless of the investment made to have the best process design, it is common that misunderstanding and misapplication of a simple 1–2-in. balance line can create a reboiler bottleneck in the entire production system.

If your plant is experiencing issues with reboilers, it is possible that the cause is related to an improper balance. It may seem almost unbelievable, but this has been the case in many reviewed reboiler installations. This article provides some basic information first, then reviews two separate applications that suffered from improper balance: 1) a de-isobutanizer (DIB) reboiler with 30,000 lb/h steam/condensate load; and 2) a de-ethanizer reboiler with 17,000 lb/h load. Explanation of how balance lines were misapplied, how that created issues, and recommendations to correct are presented.

Issues and their causes

Steam reboilers are commonly controlled with inlet steam control (ISC) for more dynamic response, or outlet condensate control (OCC) for more steady-state operation. OCC applications maintain constant steam pressure on the reboiler and adjust the reboiler duty, but ISC reboilers reduce the steam pressure to adjust temperature to the process in order to equalize the supply heat to demand heat. For the purposes of this article, focus is given to ISC applications (Figure 1) [1].

Reboilers and heat exchangers with ISC can experience multiple production and reliability issues, and mostly the causes can be attributed to either: 1) a "stall" condition [2] or 2) improper balance (Figure 2).

A stall condition occurs with ISC reboilers when the reboiler inlet steam pressure (P_2) reduces to equalize the supply heat to process heat demand and the outlet steam pressure (P_3) becomes equal to or lower than the system back pressure (P_4) (that is, $P_3 < P_4$). With either a zero or negative pressure

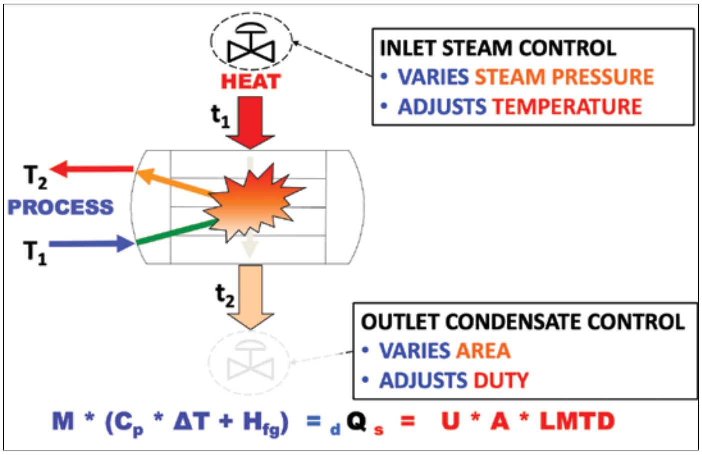

FIGURE 1. Reboiler steam or condensate controls equalize supplied heat to demanded heat by varying different factors

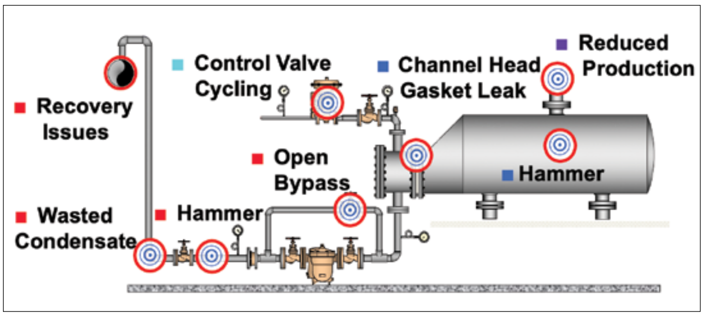

FIGURE 2. Each of the issues shown are commonly the cause of a stall or improper balance condition

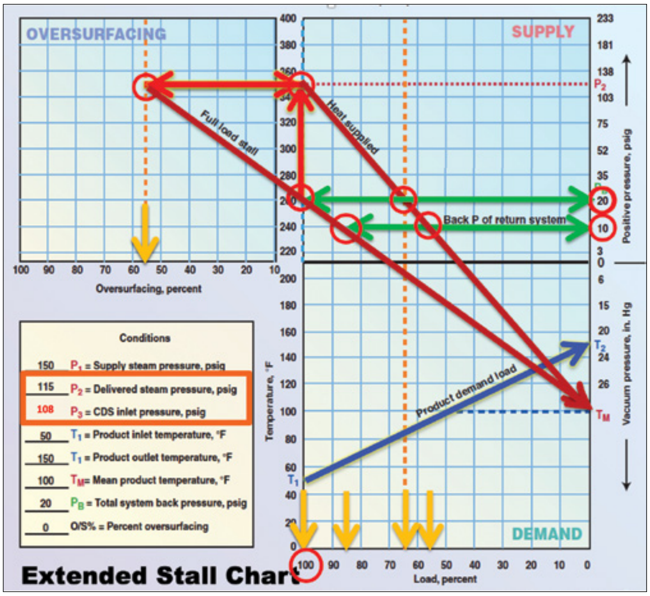

FIGURE 3. Stall points occur when the pressure profile line (maroon) intersects the back pressure line (green)

differential from the reboiler outlet (P_3) to the condensate return line (P_4), no condensate flow can occur and the system is stalled with condensate backing up into the heat-exchange surface. This condition can cause all of the issues shown in Figure 2.

An improper balance-line connection can also create a negative pressure-differential situation, but it differs in that the balanced/localized steam pressure at the condensate vessel/drum that connects to the reboiler outlet is higher than the reboiler's outlet pressure (P_3). This is explained later in the article.

Using the extended stall chart

The extended stall chart (ESC) estimates steam supply pressure for different load conditions and heat-exchange surface area (A) (from $Q = U{\cdot}A{\cdot}\Delta T$) (Figure 3). Effectively, UA "reduces" as the tube bundle becomes fouled, and the steam pressure rises to adjust supply heat to the process. The horizontal green line in Figure 3 represents equipment back pressure, and the four

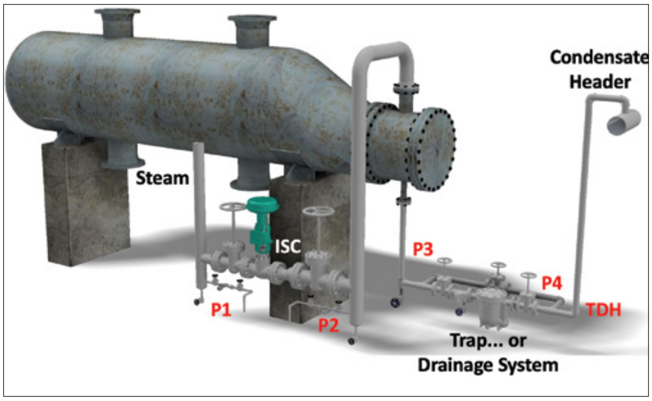

FIGURE 5. Four pressures (P_1, P_2, P_3, P_4) are needed to accurately recommend an appropriate drainage system

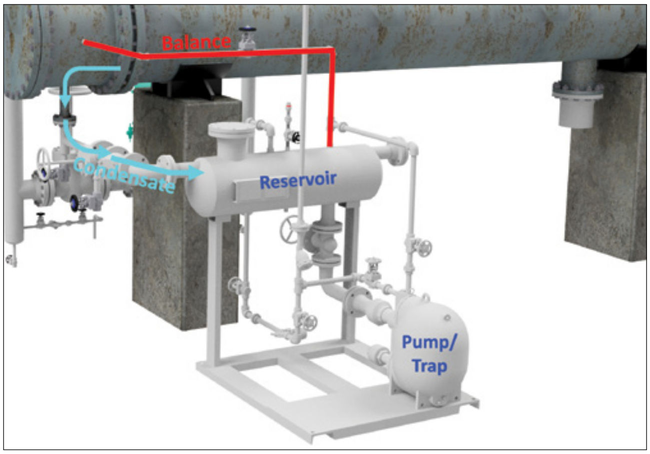

FIGURE 6. A proper condensate vessel balance line is needed so that incoming condensate can displace vapor without increasing vessel pressure

red circles where the burgundy line intersects the green line represent various individual stall points [*1, 3*].

Essentially, if the process load always maintains positive pressure differential, $P_3 > P_4$, that is, pressure is always above the back pressure line for all load conditions, then only a simple device, such as a steam trap, is needed to help drain condensate from the steam space. Alternatively, a more complex drum and outlet control valve (OCV) arrangement can be used. However, if the pressure differential is sometimes negative, $P_3 < P_4$, then another type of drainage equipment is needed, such as a combination pump/trap system. Once appropriate data are entered into the ESC calculator [*4*], the anticipated stall point is identified and a recommended drainage device can be determined (Figure 4).

Four pressures, P_1, P_2, P_3 and P_4, are needed to perform an ESC calculation (Figure 5). The main determinant of stall is the pressure differential $P_3 : P_4$. However, P_1 is needed for the design in the event that full pressure might somehow reach the drainage equipment (such as a steam trap), and the P_2 pressure at full load is critical so that the appropriate pressure profile start point can be identified on the ESC.

Often, the actual P_2 and P_3 are not known because installations may not have pressure

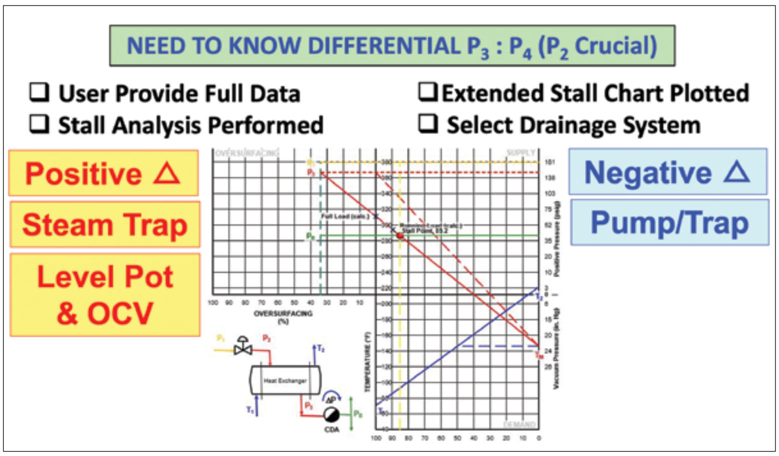

FIGURE 4. A steam trap or level pot/OCV drain system is used with positive differential pressure, and a combination pump/trap when differential pressure is sometimes negative

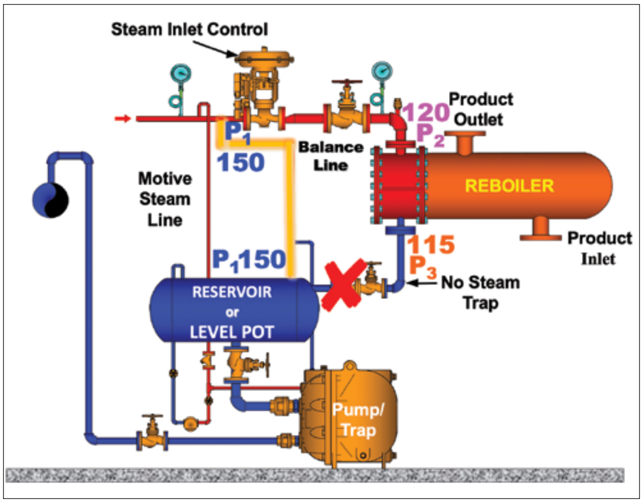

FIGURE 7. It is never recommended to balance from the P_1, inlet side pressure of the control valve

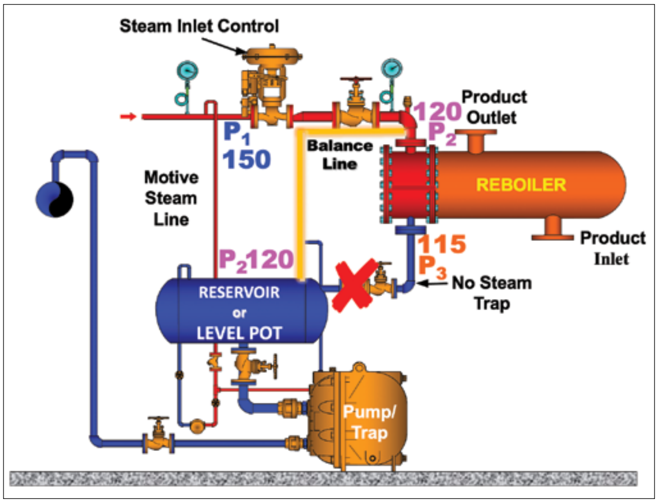

FIGURE 8. It is sometimes possible to balance from the P_2, reboiler inlet pressure, but generally not recommended

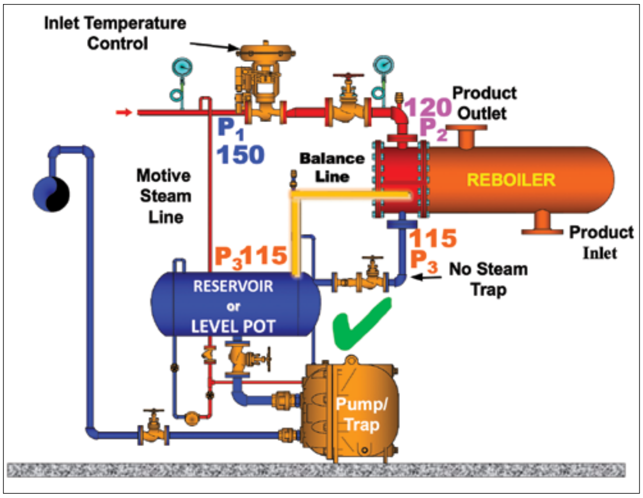

FIGURE 9. Balancing the vessel to the P_3, reboiler channel head enables unrestricted condensate flow

gages or sensors. It is crucial to know these pressures, so an appropriate method to obtain accurate readings is always recommended.

Vapor balancing basics

Balancing of a steam trap draining a reboiler is normally not necessary. However, it is required to balance either a condensate drum/level pot or reservoir when such vessels are part of the condensate drainage system.

Figure 6 provides some visualization of the need for balancing. A large body condensate vessel is often at least partially filled with steam, and when condensate enters it can compress the steam creating a higher internal pressure, thereby restricting additional condensate inflow. This can cause backup into the reboiler, which creates the issues previously outlined. When properly balanced, the vapor is displaced to the upper balance location as condensate enters the vessel body. It is important that the balance maintains the same vessel pressure as the condensate source so that flow by gravity can be accomplished.

Tube-side steam reboilers pose a special balancing challenge, and these generally horizontal installations are commonly incorrectly balanced. The red line, "Balance", in Figure 6 provides a visualization of some key aspects to balancing tube-side steam applications. In addition to avoiding low points and properly sizing the line, it is generally necessary that the line connects to the reboiler channel head, high on the outlet side after the pass partition/divider plate. In the case of combination pump/trap systems, this location is always recommended as a key requirement.

Conversely, balancing to shell-side steam reboilers is relatively easy provided that an appropriately sized tapping is located at the top side of the reboiler shell. The main items to consider are that there are no low points in the balance line where water can collect, the line is properly sized, and it is balanced to an appropriate top of shell location.

Balancing options

One practice, not recommended, is balancing the condensate vessel to the P_1 pressure located at the entrance to the ISC (Figure 7). This balances the full P_1 pressure to the vessel. P_2 represents the pressure downstream of the ISC — the pressure delivered to the reboiler, and P_3 is the outlet pressure from the reboiler — after subtracting tube pressure drop from P_2.

Even when the ISC is delivering the highest P_2, the outlet P_3 pressure of 115 psig cannot push condensate into the vessel, which is balanced to 150 psig. Balance to P_1 is expected to create a tremendous amount of backup into the reboiler, often causing operators to isolate the balance line, thereby removing balance from the system. The result of this action typically is that the vapor in the vessel becomes compressed (restricting condensate inflow), and the "work-around" is to open a bleeder valve to bleed off the pressure to atmosphere. This can create its own particular issue of pulling in air if the reboiler tube pressure goes into vacuum for low-load conditions.

Another not recommended practice is to connect the balance line to the P_2 inlet side of the reboiler (Figure 8). This brings P_2, a slightly higher pressure than P_3 (generally equivalent to or slightly higher than the tube

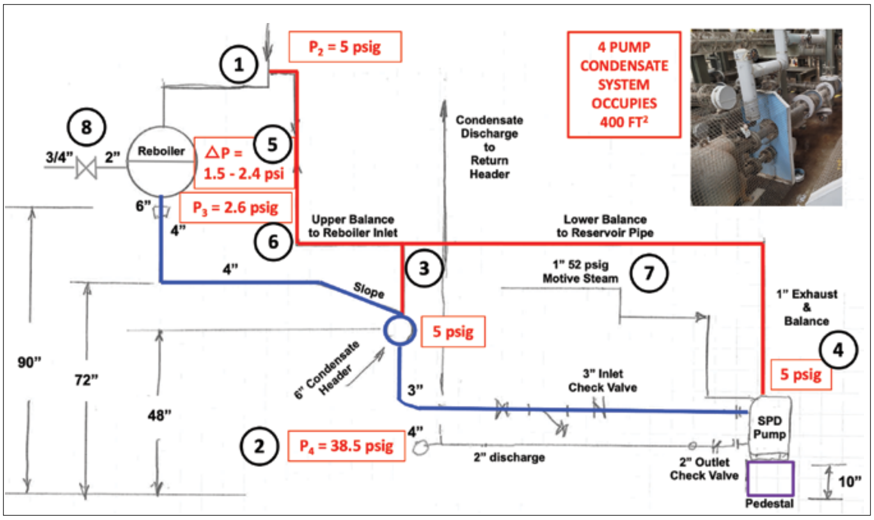

FIGURE 10. This de-ethanizer piping design had multiple hydraulic issues that prevented suitable drainage performance

pressure drop) into the condensate vessel (120 psig), against which the reboiler outlet pressure (P_3 = 115 psig) has to push to enable flow. It may work in some instances where there is sufficient fill head to create a water column high enough to overcome the pressure at the receiving vessel. In the case of this example, a water column at least 12-ft high would be needed. While this P_2 balance practice may work in some cases with level pot/OCV installations, it is not recommended for use with mechanical combination pump/trap systems.

Because drainage systems may change over time — particularly, for example, considering the potential need to use a pump/trap installation in the future when system back pressure elevates — the P_2 balance connection could require a major rework and is not recommended.

The recommended design practice for tube-side steam reboilers is to balance the condensate vessel to the outlet cavity of the channel head, on the side of the head and just below the pass partition/divider plate (Figure 9). A balance in this location brings the outlet pressure (115 psig) to the condensate vessel (115 psig) and enables flow to fill by gravity head.

De-ethanizer reboiler case review

The Figure 10 diagram provides insight into a de-ethanizer reboiler using four secondary pressure drainer (SPD) pumps/traps to drain condensate and discharge into the return header. Unfortunately, the system was improperly balanced and was not able to function, necessitating the waste of over 17,000 lb/h condensate to sewer for years (~18,000,000 gal/yr in drought area). A quick review of relative factors follows:

1. P_2 pressure = 5 psig
2. P_4 system back pressure (TDH) = 38.5 psig, so this system needs a pump solution!
3. P_2 pressure is balanced to a reservoir pipe
4. The reservoir pipe with P_2 pressure is balanced to the SPD pump
5. Reboiler high pressure drop = 2.4 psi
6. P_3 outlet pressure = 2.6 psig
7. Motive pressure to SPD pump = 52 psig, sufficient to pump against P_4 = 38.5 psig
8. The 2-in. channel head tapping is reduced to 3/4 in.

The question becomes, "Why was there difficulty when the SPD motive pressure of 52 psig was 13.5 psig higher than the back pressure (38.5 psig)?" Figure 10 explains the simple hydraulic issue that prevented this system from operating properly. The P_3 outlet pressure is 2.6 psig and the 6-in. condensate header acting as a reservoir had P_2 balance pressure of 5 psig. Condensate pushed by 2.6 psig cannot discharge into 5 psig unless the reboiler is 6 ft higher than the reservoir, but in this case, it was only 3.2 ft higher. This created a negative pressure differential, and hydraulically, the system could not drain as required.

The project engineer designed the system similar to how electric pumps could have been installed, but SPD pumps (as shown on pedestal) were used instead to avoid potential NPSHR (net positive suction head required) challenges posed by electric units. This electric pump design resulted in an installation footprint occupying over 400 ft^2 of the second level deck, and although the piping installation was of high quality, the improper balance and low-level piping removed the opportunity to pump the condensate.

There was another piping challenge to making a simple improvement to the system. The reboiler equipment designer had the correct idea and installed a 2-in. balance connection at the proper location on the channel head. However, the piping/process engineer reduced the 2-in. connection to 3/4 in. with a 3/4-in. valve instead of a 2-in. valve (Point 8 above). This made retrofit while operating a much more difficult task.

The recommended solution of a quad pump/trap package system, shown in Figure 11, occupies a much smaller 45-ft^2 footprint and could be relatively easy to install had the reboiler 2-in. channel head tap remained original size without reduction to the 3/4-in valve. Because the channel head tap was reduced, either a shutdown or hot tap on the other side of the channel head was required for retrofit. A basic recommendation is to use caution if considering to reduce the size of a channel head tap to be certain of its intended purpose and

DE-ISOBUTANIZER ISSUES*
P_2 not measured
Insufficient differential $P_3 : P_4$
Balance line size reduced
Balance line connects to P_2 steam
Insufficient differential $P_3 : P_2$
Isolation valve on balance line closed
Closure removes balance capability
Cannot back balance
No drum venting

*These factors were identified as potential causes of poor control performance

potential detriment if reduced.

In addition to the environmental impact from wasted water, it should also be noted that dumping this amount of condensate requires an additional approximate 9,300 ton/yr of steam and 1,900 ton/yr CO_2 to be produced that could be avoided if just 100°F condensate could be returned to the boiler.

De-isobutanizer reboiler case review

The two reviewed de-isobutanizers (DIB) on an alkylation column operated simultaneously, used 30,000 lb/h steam flow, and suffered from the erratic temperature control shown in the chart (top of Figure 12). There were 10 possible design-related causes to the poor performance (see box on previous page) which can be explained as follows, with reference to points shown in Figure 12:

1. P_1 pressure = varied 130–150 psig
2. P_4 system back pressure (TDH) = 43 psig, so there appears to be sufficient pressure to overcome with P_1 pressure
3. P_2 pressure = 50 psig
4. P_3 pressure = 45 psig
5. P_3 pressure confirmed by the 42–48 psig pressure reading
6. Balance line is connected to P_2, bringing P_2 pressure into the drum
7. 2-in. balance line is reduced to 1 in.
8. Balance line valve is shut off
9. The line is flooded — balance cannot occur through a flooded line
10. The system is "group-trapped" to a single drum/OCV arrangement

With a P_1 of at least 130 psig against a P_4 of 43 psig, the design team must have felt there was sufficient pressure and adequate reboiler area to accomplish the process benchmark with minimal issues. However, the system's P_3 low 42-psig pressure showed evidence of a stall condition, as well as other issues that prevented the desired performance.

Consider first that the balance line brings P_2 pressure of 50 psig to the drum, making discharge from the P_3 reboiler outlet pressure impossible at times without backing up condensate into the reboiler for needed head pressure from gravity to overcome the higher drum pressure. The site must have realized this issue because at some point they simply closed the isolation valve on the balance line, removing the 50 psig restriction to flow into the drum. However, since it is not possible to balance back to the reboiler through the flooded line routing condensate to the drum, this action then created a new issue. Once balance capability from the drum is removed, this causes a "pressure block," as the drum vapor pressure is compressed from condensate displacing some drum volume. With no place to vent, the steam (and incondensable air) is compressed to higher pressure and restricts flow until some steam condenses — or until condensate backup into the reboiler gains additional gravity fill head to overcome the higher pressure.

While not the best practice, one potential mitigating

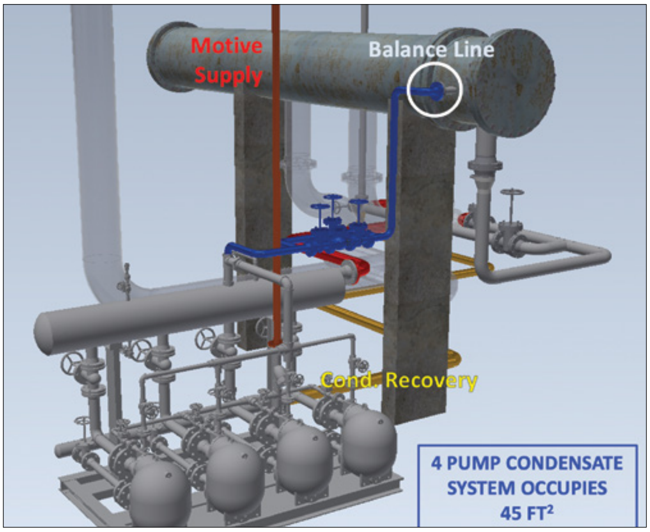

FIGURE 11. A package system mitigates piping errors and provides a much more compact drainage capability

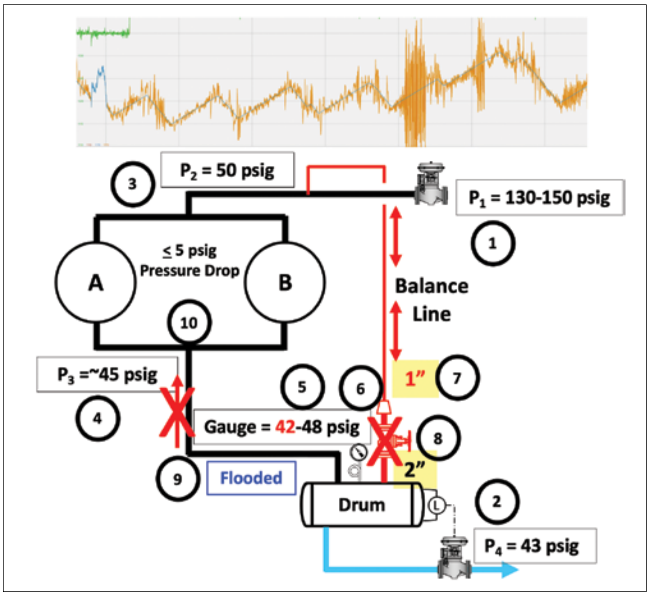

FIGURE 12. Ten potential design issues were identified that could have led to the poor temperature control

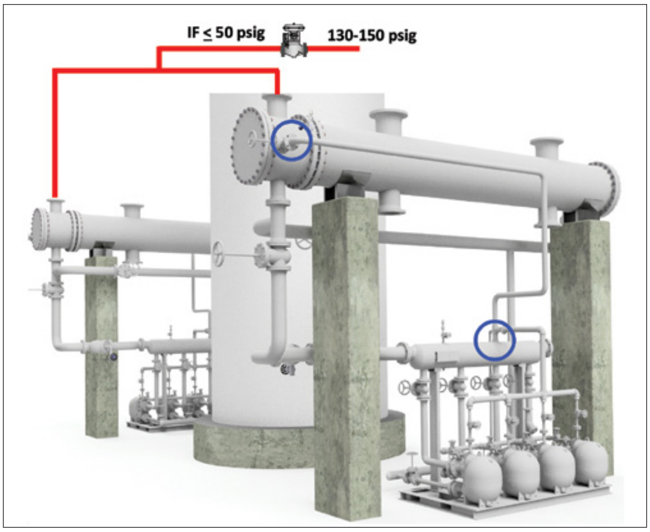

FIGURE 13. Individual quad combination pump/trap package systems can be retrofit to handle a stall condition

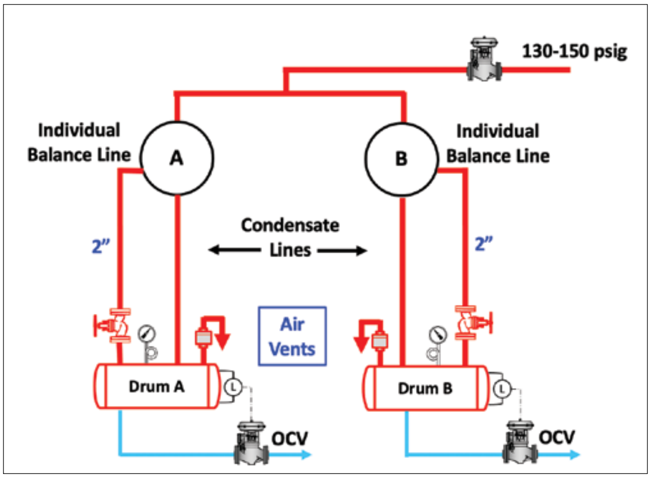

FIGURE 14. Individual condensate drum/OCV with air vents can be used in a non-stall application

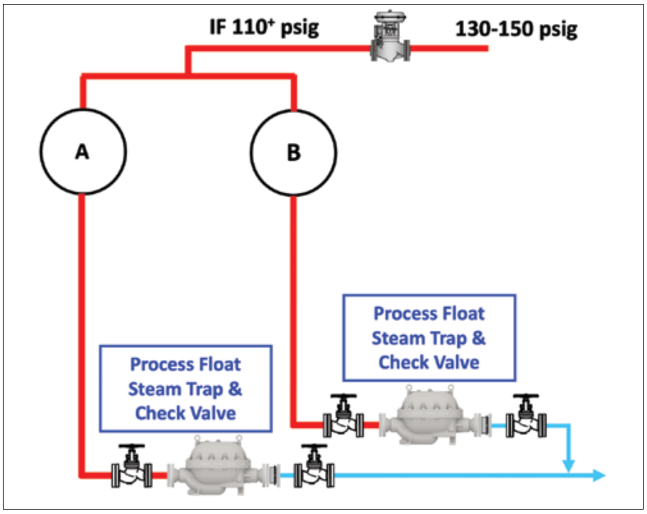

FIGURE 15. Individual steam traps with check valves are a lower cost recommendation for a non-stall application

action could have been to install a bleeder valve on the drum that could bleed off steam pressure and also incondensable air which can collect in the drum. Normally, an air vent would be recommended for a closed off system, but if a bleeder is used as a "Band-Aid" solution, then it can bleed both air and steam.

Even if the system had been properly balanced to the outlet side of the channel head just below the pass partition/divider plate, there was an additional issue of "group trapping" [*5*], indicated by Point 10 on Figure 12. This occurs when two or more condensing sources are discharged into a single condensate vessel or steam trap. Although the reboilers may look identical, there is commonly a different rate of condensation internally, which causes one of the reboiler's outlet pressure to be higher than the other. The higher outlet pressure becomes a restriction to the other's flow, thereby causing backup and erratic temperature control in that reboiler. This is why group-trapping condensing equipment is almost never recommended.

Because the equipment experienced stall conditions, quad combination pump/trap package systems were recommended. These require a proper channel head tapping for balance (as reviewed previously in the article), which fortunately existed on the reboilers with full size isolation valves to make the installation relatively simple to accomplish. To avoid the issues of group trapping, individual quad pump/trap packages were recommended for each DIB, with proper balance to the channel head (Figure 13).

Had the reboilers sufficient positive differential pressure ($P_3 > P_4$) and not been experiencing a stall condition, individual condensate drums with OCV could have been a recommended option, properly balanced to the channel head and with air vents installed on each drum (Figure 14). This would have been a costlier option than needed, because with positive $P_3 > P_4$ pressure differential, simple float-style steam traps and check valves would be an easier and less costly recommendation (Figure 15).

Closing thoughts

Properly locating a simple 1– 2-in. balance line can make all the difference in successful reboiler operation. Paying close attention to this detail during the design phase of a project can yield benefits throughout the entire operational life of the distillation column. ■

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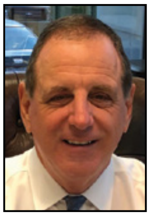
Author



James R. Risko is retired from TLV Corp. (13,901 South Lakes Dr., Charlotte, N.C. 29873; Phone: 704-641-8959; Email: jimrisko@gmail.com), where he was president and responsible for U.S. and Canadian operations. He has over 47 years' steam-systems experience, authored more than 60 technical articles, provided webinars to over 3,500 attendees globally, and presented papers for the Kister Distillation Symposium, Distillation Experts Conclave, Fractionation Research Inc., American Fuel & Petrochemical Manufacturers (AFPM), American Institute of Chemical Engineers (AIChE), the Ethylene Conference, Refing Community (RefComm), International Pressure Equipment Integrity Assn. (IPEIA), IETC, eChemExpo, Assn. of Energy Engineers (AEE) World and American Society of Heating, Refrigerating and Air-Conditioning Engineers (ASHRAE). He co-invented the world's first combination pump/trap and created the "extended stall chart" for draining stalled coils, heat exchangers and reboilers, the "drop-down loop seal" concept to help mitigate hammer in vertical risers of flashing condensate lines, and the two-bolt combined steam trap strainer-connector. A past chairman of the FCI, he has been selected to receive its 2024 Lifetime Achievement Award. Risko is currently an Advisory Board member of both the Texas Industrial Efficiency Energy Program (TIEEP) and the TEES Industrial Energy Technology Conference (IETC), an avid tennis and guitar player, and has three energy management certifications. He holds an M.B.A from Wilkes University, and two B.S. degrees, in mathematics/education and business administration/accounting, from Kutztown University.

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Optimization of Spray Drying with Exhaust-Gas Recirculation

Energy recovery in spray-drying processes can reduce production costs, but involves trade-offs. Using milk powder as an example, this article describes a spreadsheet method to evaluate exhaust-gas recirculation scenarios for optimizing spray drying

Yick Eu Chew, Wei Wen Wee, Timothy Gordon Walmsley and Dominic C.Y. Foo
University of Nottingham, Malaysia
University of Waikato, New Zealand

Dairy processing is among the most energy and carbon intensive operations in the global food processing industry. Estimates say dairy farms are responsible for 4.4% of total anthropogenic greenhouse gas (GHG) emissions, which is approximately 2.1 Gt/yr of carbon dioxide equivalent (CO_{2e}) [*1, 2*]. Within dairy processing, cheese production has the highest energy requirement (8.33 MJ/kg product) [*3*], followed by milk powder processing (4.60 MJ/kg product) [*4*]. The large energy requirements in milk powder production are primarily due to the drying and evaporation processes.

With improvements in energy efficiency, it is predicted that the energy consumption of the global dairy processing sector could be reduced by 50 to 80% — equivalent to 1.05–1.68 Gt/yr of CO_{2e} [*5*]. Therefore, the food processing industry has increased attention on energy recovery and heat integration of drying processes as a main priority in efforts to save energy and reduce production costs [*6*].

One example of this energy recovery is exhaust-air recirculation for a dairy spray-drying process. In this article, we explain the concepts of exhaust-air recirculation and discuss how to create a nearly closed-loop configuration. For milk spray drying, the recirculation of exhaust gas will lead to complex energy trade-offs between heat recovery and additional hot utility to dehumidify the exhaust air and regenerate its drying capacity. Through optimization of a validated model, we are able to identify optimum recycle ratios that minimize total utility use and estimated cost.

Process description

Drying systems in food manufacturing factories can produce a range of milk powder grades, such as whole- and skim-milk powder. The system considered here includes a tall form dryer, as well as a spray dryer connected to a fluidized-bed dryer (Figure 1). To enable exhaust recirculation, we investigated the addition of a dehumidification system and a wet scrubber. For the experiments, it was assumed that the plant was located in Malaysia and has an annual operating time (AOT) of 6,000 hours.

Ambient air at 30°C with a relative humidity of 0.0211 $kg_{water}/kg_{dry\ air}$ mixes with the recirculated air and enters the adsorber. Post drying, silica gel dehumidifies the process air (including the recirculated exhaust air), before being sent for the drying process of milk concentrate. The silica gel releases the bound water and regenerates as hot, dry air passes through it.

The integrated spray-dryer and fluidized-bed dryer system produces powder creamer of low dairy content from milk concentrate. In the spray-drying chamber, atomized liquid droplets meet hot air at 160°C with a relative humidity of 0.016 $kg_{water}/kg_{dry\ air}$ to become creamer. The remaining material enters the fluidized-bed dryer with a moisture content of 0.06 kg H_2O per kg of creamer. Note that the drying activity mainly takes place in the spray-drying chamber, while the fluidized-bed dryer mainly cools the product and prevents the powder from agglomerating excessively.

On the other hand, the tall form dryer produces lower-grade milk powder from concentrate. In its drying chamber, atomized milk droplets come into contact with hot air at 180°C with a relative humidity of 0.016 $kg_{water}/kg_{dry\ air}$. The difference in temperature profile for both

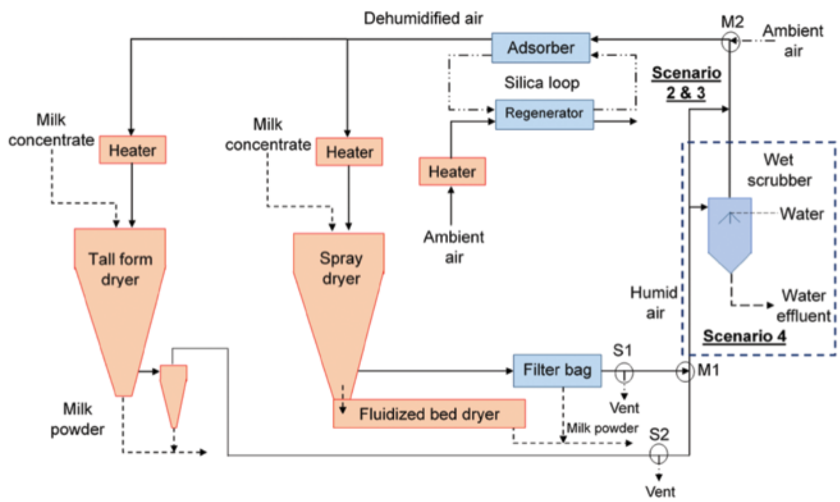


FIGURE 1. The diagram shows the spray-drying system discussed here, with exhaust-gas recirculation for different scenarios

Scenario	Recycling ratio 1, R_1 (%)	Recycling ratio 2, R_2 (%)	Annual savings on heater duty ($/yr)
1 (base case)	0	0	-
2	100	18	43,339
3	100	2	37,843
4	59	0	4,566

TABLE 1. Optimum recycling ratio for each scenario and annual savings on heater duty

dryers results in products with different qualities. The final moisture content of the milk powder exiting the tall form dryer is 0.03 kg H_2O per kg of powder. The exhaust gas from the spray dryer and tall form dryer passes through a filter bag and cyclone to recover suspended powder particles.

Case study

In this work, we look at three concepts for the recirculation of exhaust gas with comparison with the current process operation.

- Base case (no exhaust-gas recirculation)
- Recirculation of exhaust gas while considering the humidity limit of drying air only
- Recirculation of exhaust gas while considering the humidity limit of drying air and fines limit of exhaust gas
- Recirculation of exhaust gas passes through a wet scrubber its fine particles are removed

Optimization model

Scenario 1 describes the base case without exhaust gas recirculation. The total energy consumption (Q_{total}) of the heaters was calculated using Equation (1). The unit price of electricity (C_{elec}) is $0.08/kWh based on a typical industrial tariff [7]. The total cost (C_{total}) for heaters duty is calculated by using Equation (2). The base case serves as a benchmark for subsequent scenarios.

$$Q_{total}\ (\text{kW/h}) = Q_{SD} + Q_{TD} + Q_{reg} \quad \textbf{(1)}$$

$$C_{total}\ (\$/\text{yr}) = Q_{total} \times C_{elec} \times AOT \quad \textbf{(2)}$$

Where Q_{SD} is the heat duty for spray dryer, Q_{TD} is the heat duty for tall form dryer and Q_{reg} is the regenerative duty. The result shows that the total heat duty is 498 kW, with a cost of $241,708 per year.

Scenario 2 describes the system when part of the exhaust gas from the spray dryer and tall form dryer is recirculated. However, the process air for the dryers needs to have an absolute humidity ($y_{ada,out}$) of below 0.016 $kg_{water}/kg_{dry\ air}$ to ensure efficient drying. By implementing the model in Microsoft Excel as shown in Figures 2 and 3, it is easy to investigate the effects of the two recycle ratios and minimize the overall energy consumption of the system:

$$min\ (Q_{total}) \quad \textbf{(3)}$$

The critical variables to optimize include the air-recycling ratios for spray dryer (R_1) and tall form dryer (R_2), and the temperature for regeneration of silica gel ($T_{Ra,in}$). Because the model is linear, the Simplex (standard method of maximizing or minimizing a linear function of several variables under several constraints on other linear functions) LP method using Microsoft Excel can quickly minimize the objective function (Equation 3) given the following constraints:

$$0 \le y_{ada,out} \le 0.016\ kg_{water}/kg_{dry\ air} \quad \textbf{(4)}$$

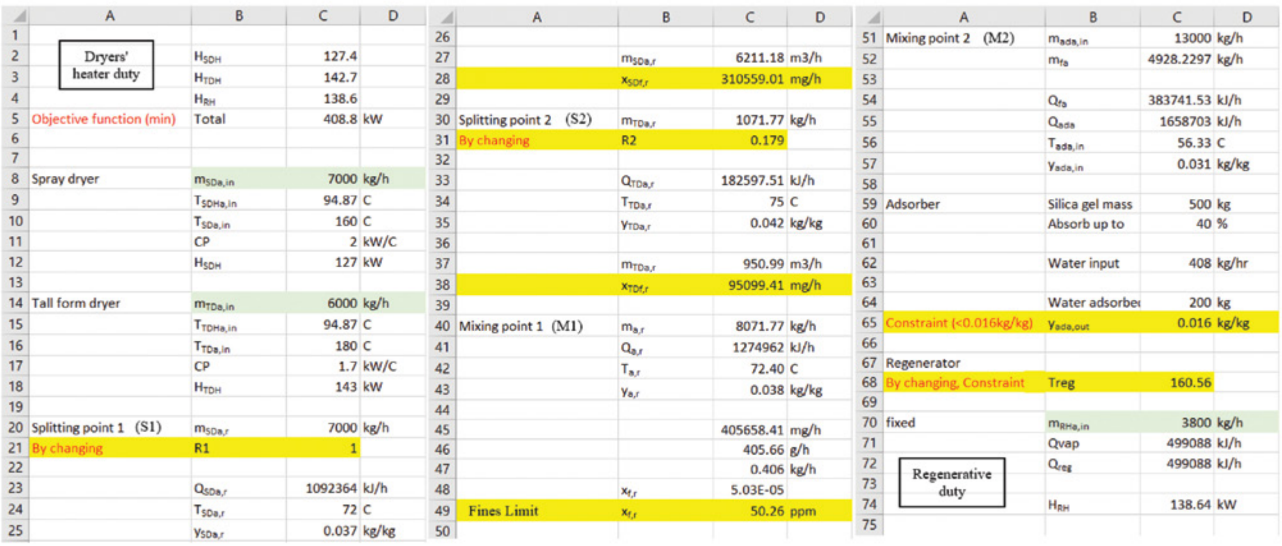
$$150 \le T_{Ra,in} \le 180^{\circ}C \quad \textbf{(5)}$$

$$0 \le R_1 \le 1 \quad \textbf{(6)}$$

$$0 \le R_2 \le 1 \quad \textbf{(7)}$$

Figure 4 illustrates the effect of recycling ratios on the temperature input into the dryer heaters. It shows that with the recirculation of exhaust gas, the inlet temperature to the main air heaters increases, leading to less hot-utility duty to supply air at the required operating setpoints. Figure 5 also shows the effect of the recycling ratios on the regeneration heater duty.

As R_1 increases from 20% to 80%, the duty for dryer heaters decreases by as much as 16%. On the other hand, as R_2 increases from 0% to 60%, the dryer heaters' duty reduces by up to 15%. However, as R_1 and R_2 increase, the regenera-



	A	B	C	D
1				
2	Dryers' heater duty	H_{SDH}	127.4	
3		H_{TDH}	142.7	
4		H_{RH}	138.6	
5	Objective function (min)	Total	408.8	kW
6				
7				
8	Spray dryer	$m_{SDa,in}$	7000	kg/h
9		$T_{SDHa,in}$	94.87	C
10		$T_{SDa,in}$	160	C
11		CP	2	kW/C
12		H_{SDH}	127	kW
13				
14	Tall form dryer	$m_{TDa,in}$	6000	kg/h
15		$T_{TDHa,in}$	94.87	C
16		$T_{TDa,in}$	180	C
17		CP	1.7	kW/C
18		H_{TDH}	143	kW
19				
20	Splitting point 1 (S1)	$m_{SDa,r}$	7000	kg/h
21	By changing	R1	1	
22				
23		$Q_{SDa,r}$	1092364	kJ/h
24		$T_{SDa,r}$	72	C
25		$y_{SDa,r}$	0.037	kg/kg
26				
27		$m_{SDa,r}$	6211.18	m3/h
28		$x_{SDf,r}$	310559.01	mg/h
29				
30	Splitting point 2 (S2)	$m_{TDa,r}$	1071.77	kg/h
31	By changing	R2	0.179	
32				
33		$Q_{TDa,r}$	182597.51	kJ/h
34		$T_{TDa,r}$	75	C
35		$y_{TDa,r}$	0.042	kg/kg
36				
37		$m_{TDa,r}$	950.99	m3/h
38		$x_{TDf,r}$	95099.41	mg/h
39				
40	Mixing point 1 (M1)	$m_{a,r}$	8071.77	kg/h
41		$Q_{a,r}$	1274962	kJ/h
42		$T_{a,r}$	72.40	C
43		$y_{a,r}$	0.038	kg/kg
44				
45			405658.41	mg/h
46			405.66	g/h
47			0.406	kg/h
48		$x_{f,r}$	5.03E-05	
49	Fines Limit	$x_{f,r}$	50.26	ppm
50				
51	Mixing point 2 (M2)	$m_{ada,in}$	13000	kg/h
52		m_{fa}	4928.2297	kg/h
53				
54		Q_{fa}	383741.53	kJ/h
55		Q_{ada}	1658703	kJ/h
56		$T_{ada,in}$	56.33	C
57		$y_{ada,in}$	0.031	kg/kg
58				
59	Adsorber	Silica gel mass	500	kg
60		Absorb up to	40	%
61				
62		Water input	408	kg/hr
63				
64		Water adsorbe	200	kg
65	Constraint (<0.016kg/kg)	$y_{ada,out}$	0.016	kg/kg
66				
67	Regenerator			
68	By changing, Constraint	Treg	160.56	
69				
70	fixed	$m_{RHa,in}$	3800	kg/h
71		Qvap	499088	kJ/h
72	Regenerative duty	Q_{reg}	499088	kJ/h
73				
74		H_{RH}	138.64	kW
75				

FIGURE 2. A Microsoft Excel spreadsheet can be used to determine optimum exhaust-gas recycling ratios for the spray-drying system

tion duty increases as well. When R_1 increases from 20% to 80% and R_2 from 0% to 60%, the regeneration heater duty increases by 2.3% and 2.1%. As R_1 and R_2 increase, the absolute humidity of the air into the adsorber increases, leading to a faster saturation of the silica gel. As a result, the silica gel requires additional hot air from a heater to regenerate. For this specific case, the optimization suggests that that the entire exhaust gas from the spray dryer (R_1) and 18% of the exhaust gas from the tall form dryer (R_2) should be recycled. This optimum combination of recycling ratio has a total duty of 409 kW with an annual cost of $198,369.

Scenario 3 assumes the adsorber has a fines limit ($x_{f,r}$) of 45 parts per million (ppm). As a result, the amount of fines within the recirculated gas should be limited. An additional constraint in the model, Equation (8) provides for the fines limit. The exhaust gas from cyclone and filter bag is assumed to contain 100 mg/m^3 and 50 mg/m^3 fines.

$$0 \leq x_{f,r} \leq 45 \text{ ppm} \qquad (8)$$

Now the objective in Equation (3) can be minimized subject to the constraints in Equations (4) to (8). In this case, the optimum recycling ratio obtained is total recirculation from the spray dryer (R_1) and 2% from the tall form dryer (R_2). Under these recirculation conditions, the heater duty reduces to 420.10 kW. The total cost for heater duty is $203,865/yr.

Scenario 4 proposes a wet scrubber to actively remove fines in the exhaust gas. The wet scrubber has an estimated removal efficiency of 90% for a liquid-to-gas ratio (L/G) for the wet scrubber of 0.94 kg/m^3 [8]. A final assumption is that the wet scrubber operates adiabatically.

Last of all, the model can be rerun to achieve the condition in Equation (3) subject to the constraints in Equations (4) to (8) and a fines upper limit in the recirculated air of 4 ppm. The optimum recycling ratio now reduces to 59% from the spray-dryer exhaust air (R_1) and no recirculation from the tall form dryer (R_2). Although the purpose of the wet scrubber is to remove fine particles to increase the recycle ratio, the result shows that the maximum recycling ratio decreases with the introduction of a wet scrubber. This is because when the exhaust gas passes through the wet scrubber, it adds water to the air, increasing its absolute humidity and decreasing its temperature drastically due to water evaporation [9]. Due to the high absolute humidity of the wet scrubber outlet, and the constraint of process air absolute humidity of less than 0.016 kg water/kg dry air, the recycling ratio decreases in Scenario 4 compared to the other two recirculation scenarios. For Scenario 4, the heater duty is evaluated to be 489 kW. On the other hand, the total cost for heater duty is calculated to be $237,142/yr.

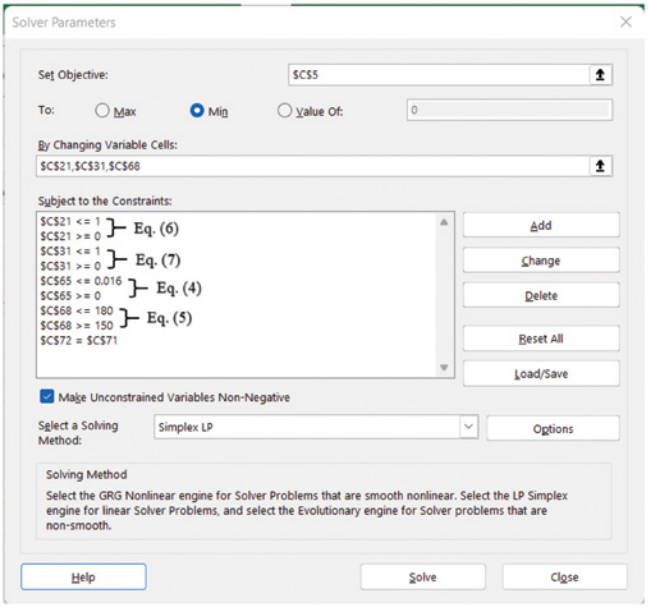


FIGURE 3. The figure shows the set up of the Excel solver to minimize the heat duty, subjected to constraints

Comparison of results

The results from the base-case model and those from the different scenarios are shown in Figure 6.

The required heater duty decreases as the inlet temperature increases, due to the increase in recycling ratio. The regeneration heater duty remains stagnant throughout four scenarios, as presented in the bar chart. On the other hand, there is an obvious decreasing trend for dryer-air heater duty compared with the base-case model. As a result, the increased regeneration heater duty is not as significant as the decrease in dryer heaters duty. Moreover, as heater duty is depending on the electricity cost, both the total heater duty and annual heater cost have a similar trend. The optimum recycling ratios that yield the lowest total duty are summarized in Table 1.

To conclude, we have presented four scenarios — three dryer-air recirculation scenarios and the current system design without recirculation (Scenario 1). Scenario 2 achieves the lowest heater duty, corresponding to an 18% reduction, with annual

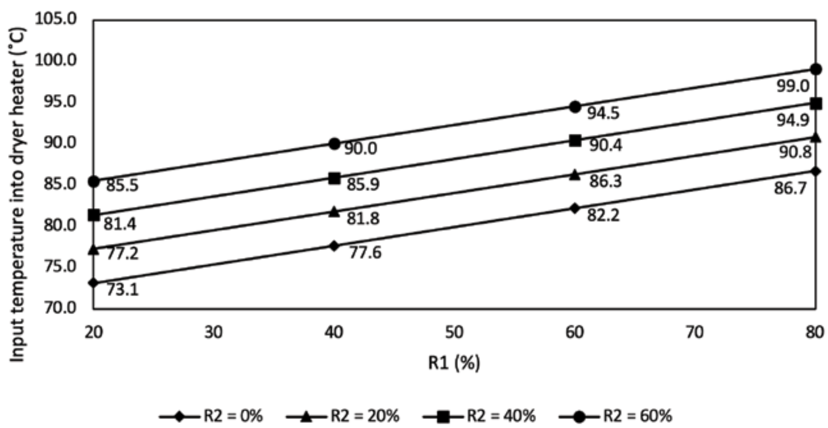
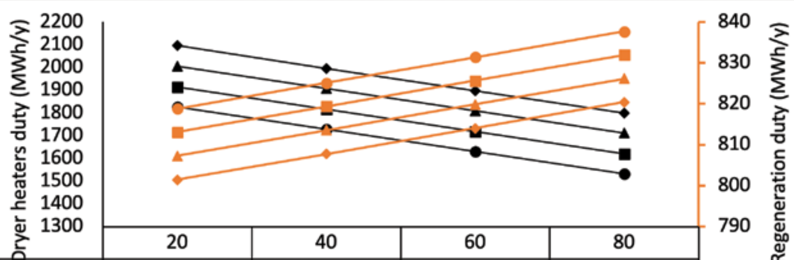


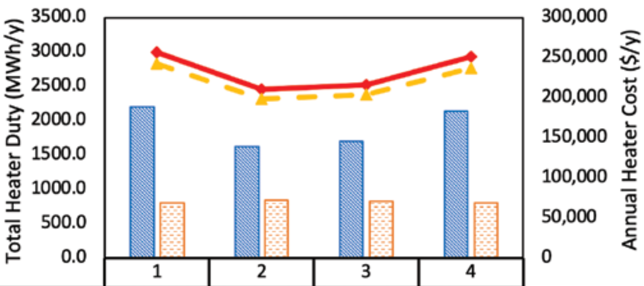
FIGURE 4. This graph illustrates the relationship between reycling ratios and the inlet temperature of the dryer heater



	20	40	60	80
R2 = 0%	2095	1996	1898	1799
R2 = 20%	2005	1907	1808	1710
R2 = 40%	1915	1817	1718	1620
R2 = 60%	1825	1727	1628	1530
R2 = 0%	801	808	814	820
R2 = 20%	807	814	820	826
R2 = 40%	813	819	826	832
R2 = 60%	819	825	831	838

R1 (%)

FIGURE 5. The graph and associated table show the relationship between recycling ratios and dryer and regeneration heater duties



	1	2	3	4
Dryer air heater duty (MW/y)	2193.3	1620.8	1693.4	2130.6
Regeneration heater duty (MW/y)	795.1	831.8	827.1	801.5
Total heater duty (MW/y)	2988.5	2452.6	2520.6	2932.0
Annual heater cost ($/y)	241,708	198,369	203,865	237,142

FIGURE 6. The total heater duty for each optimized scenario is shown here

savings of $57,785/yr. However, the fines in the dryer exhaust air streams may clog the adsorber and lead to a reduction in energy efficiency in the long run. In Scenario 3, a constraint for the fines limit was applied and the reduction in annual heater duty and savings was determined to be 16% and $50,457/yr. The total elimination of fines in the exhaust air is proposed to enable an energy-efficient, total closed-loop drying system. In Scenario 4, the introduction of a wet scrubber to eliminate fines was analyzed. The annual heater-duty decreases was 2%, but it was not enough to compensate for the cost of water required for the wet scrubber unit. This is because, with the assumptions of L/G ratio of 0.94 kg/m^3, the water flowrate required is 3,457 kg/h, which leads to high effluent disposal charges. The freshwater tariff at the location of the plant (Johor, Malaysia) is $0.81 per ton of water. As a result, the cost of water required for the wet scrubber adds up to $22,402 annually, which is not enough to be covered by the savings from the decrease in heater duty ($6,087 per year). ■

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Authors

Yick Eu Chew, is a chemical engineering graduate from the University of Nottingham Malaysia (B34, 43500 Semenyih, Selangor, Malaysia). He is now undertaking postgraduate study at Swinburne University of Technology Sarawak Campus. His research works focus on process design and optimization.

Wei Wen Wee is a chemical engineering graduate from the University of Nottingham Malaysia (www.nottingham.edu.my). Her research interests are in process optimization, waste treatment and management.

Timothy Gordon Walmsley is a senior lecturer in process and energy engineering in the School of Engineering and assistant director of the Ahuora Centre for Smart Energy Systems at the University of Waikato in New Zealand. He is a chartered engineer and member of the Institution of Chemical Engineers (IChE).

Dominic C. Y. Foo is a professor of process design and integration at the University of Nottingham Malaysia (Dominic.Foo@nottingham.edu.my). He is a past president of the Asia-Pacific Confederation of Chemical Engineering, a fellow of the Academy of Sciences Malaysia and the Institution of Chemical Engineers.

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Economic Indicators

2021 2022 2023

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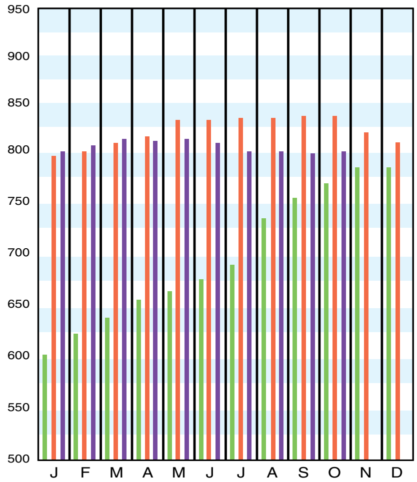
CHEMICAL ENGINEERING PLANT COST INDEX (CEPCI)

(1957–59 = 100)	Oct. '23 Prelim.	Sept. '23 Final	Oct. '22 Final
CE Index	790.8	793.3	816.2
Equipment	992.0	995.7	1,034.4
Heat exchangers & tanks	808.0	812.8	865.7
Process machinery	1,016.0	1,021.8	1,040.4
Pipe, valves & fittings	1,329.7	1,330.2	1,462.3
Process instruments	560.5	562.2	549.6
Pumps & compressors	1,484.4	1,484.4	1,321.8
Electrical equipment	802.7	801.8	781.1
Structural supports & misc.	1,103.4	1,112.5	1,160.6
Construction labor	375.1	374.6	362.4
Buildings	801.0	808.0	807.8
Engineering & supervision	315.7	313.3	311.6

Annual Index:
2015 = 556.8
2016 = 541.7
2017 = 567.5
2018 = 603.1
2019 = 607.5
2020 = 596.2
2021 = 708.8
2022 = 816.0

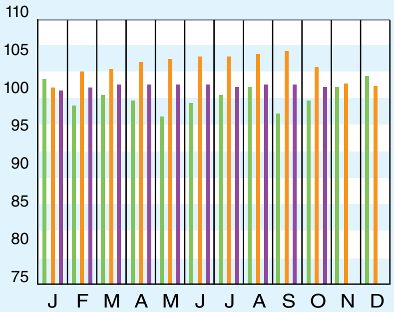


Starting in April 2007, several data series for labor and compressors were converted to accommodate series IDs discontinued by the U.S. Bureau of Labor Statistics (BLS). Starting in March 2018, the data series for chemical industry special machinery was replaced because the series was discontinued by BLS (see *Chem. Eng.*, April 2018, p. 76–77.)

CURRENT BUSINESS INDICATORS

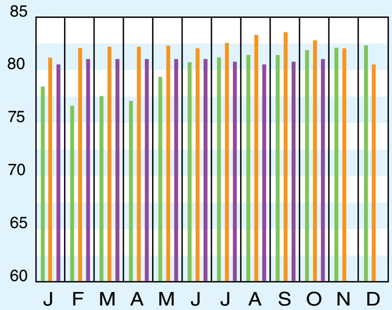
CURRENT BUSINESS INDICATORS	LATEST	PREVIOUS		YEAR AGO
CPI output index (2017 = 100)	Oct. '23 = 99.7	Sept. '23 = 100.2	Aug. '23 = 99.3	Oct. '22 = 100.6
CPI value of output, $ billions	Sept. '23 = 2,478.8	Aug. '23 = 2,446.1	Jul. '23 = 2,373.1	Sept. '22 = 2,515.4
CPI operating rate, %	Oct. '23 = 79.3	Sept. '23 = 79.7	Aug. '23 = 79.1	Oct. '22 = 80.8
Producer prices, industrial chemicals (1982 = 100)	Oct. '23 = 318.2	Sept. '23 = 309.2	Aug. '23 = 307.5	Oct. '22 = 341.7
Industrial Production in Manufacturing (2017 =100)*	Oct. '23 = 99.0	Sept. '23 = 99.7	Aug. '23 = 99.5	Oct. '22 = 100.8
Hourly earnings index, chemical & allied products (1992 = 100)	Sept. '23 = 226.6	Aug. '23 = 224.3	Jul. '23 = 224.9	Sept. '22 = 207.8
Productivity index, chemicals & allied products (1992 = 100)	Oct. '23 = 91.2	Sept. '23 = 91.6	Aug. '23 = 91.0	Oct. '22 = 91.4

CPI OUTPUT INDEX (2017 = 100)†



CPI OUTPUT VALUE ($ BILLIONS)

CPI OPERATING RATE (%)



*Due to discontinuance, the Index of Industrial Activity has been replaced by the Industrial Production in Manufacturing index from the U.S. Federal Reserve Board.
†For the current month's CPI output index values, the base year was changed from 2012 to 2017
Current business indicators provided by Global Insight, Inc., Lexington, Mass.

CURRENT TRENDS

The preliminary value for the *CE* Plant Cost Index (CEPCI; top) for October 2023 (most recent available) is lower than the previous month's value, continuing a trend of lower monthly values that has been observed since May 2023. To arrive at the overall CEPCI decline for October 2023, decreases in the Equipment and Buildings subindices offset small increases in the Construction Labor and Engineering & Supervision subindices. The current CEPCI value now sits at 3.1% lower than the corresponding value from October 2022. Meanwhile, the Current Business Indicators (middle) show small decreases in the CPI output index and the CPI operating rate for October 2023, and an increase in the CPI value of output for September 2023.